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1. Representing the Lincolnshire Iron Masters Association, or member firms.
2. Representing the Midland Association of Blast Furnace Owners, or member firms.
3. Representing the Northamptonshire Blast Furnace Owners Association, or member firms.
4. Independent Members.
5. Representing the South Yorkshire Survey Committee of the Fuel Research Board.
6. Representing the South Yorkshire Coal Trade Association.
7. Representing the Coke Oven Managers Association.
8. Co-ordinating members.
9. Representing the University of Sheffield.

For convenient discussion of the work carried out, the Committee is subdivided into two sections, namely, the Coke Users (Metallugical) Section, of which the writer is Chairman, and the Coke Makers Section, of which Mr. J. Brass is Chairman. The broad lines of research are agreed upon by the Committee, acting in conjunction with the Coke Co-ordinating Committee, representing the Midland, Northern and Scottish Coke Research Committees, with Dr. F. S. Sinnatt as Chairman. The direction of the research is in the hands of Professor Wheeler and Dr. Mott and is carried out in the laboratories of the Department of Fuel Technology of Sheffield University, at the experimental oven plant of the Committee installed at the coke-oven plant of the Tinsley Park Colliery Co., Ltd., and at coke-oven and blast-furnace plants of members of the Committee. The results of the research work are circulated to members of the Committee (and also to members of the Northern and Scottish Coke Research Committees) as confidential progress reports at least half-yearly.

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The staff of the Midland Coke Research Committee responsible for most of the experimental work described in this Report have been:

J. Hiles, M.Sc., A.I.C.	General Testing and Research.
D. F. Marshall, B.Sc.Tech., Ph.D.	Blast Furnace tests, Experimental oven tests (to 1936).
C. E. Spooner, B.Sc., M.Sc.Tech., A.I.C.	Laboratory study of the coking properties of coals (since 1935).
W. Brewin, Assoc. Fuel	Combustion of coke, photography.
J. Hodgkinson	Experimental oven tests.
F. Frith	Physical tests on coke.
W. Thompson	Assistant to Mr. J. Hiles since 1935.

M. Beevor (1929–1932) and F. Smith (1932–1934) were concerned with laboratory coking tests on the resignation of Dr. R. G. Davies (1928–1931) from the staff.

R. W. Perry (1927–1933) was concerned with experimental oven tests.

In addition, the thanks of the Committee are due to: J. Allinson, M.Sc., Ph.D.; R. Belcher; L. Burdekin, M.Sc.Tech.; F. Heathcoat, M.Sc., Ph.D.; J. K. Thompson, M.Sc., B.Sc.Tech., Ph.D., for their voluntary assistance.

(*Signed on behalf of the Midland Coke Research Committee*)

W. J. Brooke (*Chairman*).

November, 1938.

PREFACE

In the First Report of the Midland Coke Research Committee, "Coke for Blast Furnaces," published in 1930, we reported on the results of work completed up to the end of 1929. The present Report deals with work completed by the end of 1937, with an occasional summary, where it seemed essential, of work carried out subsequently.

When preparing a report of this nature it is necessary to plan the principal chapters of the book and to fit into them, where most convenient, the results of individual branches of the work. Since the complete programme of the Committee is comprehensive, and work on most of the subjects in it has been undertaken, the records of some of the subjects studied cannot conveniently be fitted into the present Report. This is so with regard to work on the use of coke for domestic purposes. We have dealt briefly with this subject in Chapter XI, under the heading "The Reactivity of Coke," but a more comprehensive report on this subject alone is needed. Work which has been omitted is that on the relationship between the composition of a coal and the quality of its volatile matter, on the classification of coal, and on the testing of coals liable to cause damage to oven walls.

All the work reported here has formed the subject of progress reports to the Committee, and some of it has subsequently been used in papers for the technical press. We trust that the treatment now accorded this work will further the principal object of the Committee, namely, extension of knowledge of the factors which influence the quality of coke. Since the principal factor which influences the quality of coke is the quality of the coal used for making it, the composition of coal, the relationship between the composition and properties of coal, and the fundamental factors of coke-formation have been dealt with at considerable length.

We have been able to demonstrate the influence of the composition of coal on the quality of the coke made from it, and to show the valuable effect of the presence of both durain and fusain. We have been able to define fairly precisely the advantages of blending with coke dust. But we are still baffled by the inter-reactions which take place between one (bright) coal and another, and we are unable to draw more than tentative conclusions as to the value of blending two or more such coals in different proportions.

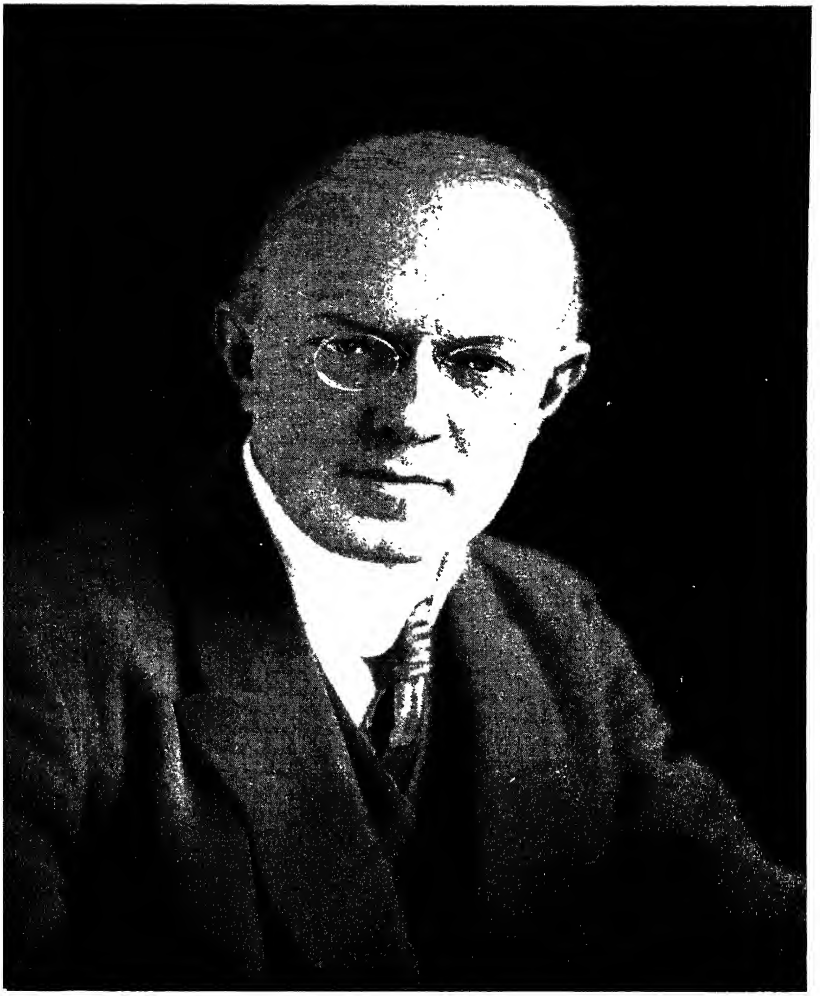
The full value of work carried out by research organizations can

only be realised when the results of that work have been applied. Whilst we believe that our treatment of the inter-relationships of the properties of a coal with its composition, and of one property with another, which may be said to be the general thesis of this Report, will improve general understanding of the problems associated with the quality of coke, we have paid particular attention to the standardizing of methods of testing which may be employed by others. When applying such methods it is important to realise the effect of the errors inherent in them, and it has been necessary to discuss fully the application of the theory of errors. Although the theory of errors has been applied in particular to the results of the "shatter test," the methods of calculation may be applied to any other series of values, and we would particularly urge the importance of the theory, the application of which to the standardization of methods of sampling and testing coal is due to a member of the Committee, Dr. E. S. Grumell.

We have acknowledged in the appropriate places our indebtedness to other workers. We wish to acknowledge here our appreciation of the skill of two members of the staff of the Midland Coke Research Committee, Messrs. J. Hiles and W. Brewin, who have been responsible, the one for the line drawings, and the other for the photographs for the half-tone blocks. Whilst special acknowledgment is thus made, the Report owes much to the enthusiasm of each member of the staff of the Midland Coke Research Committee and to the voluntary assistance of members of the Department of Fuel Technology of Sheffield University, who have adopted faithfully the motto of their University, "Rerum Cognoscere Causas."

R. A. Mott.
R. V. Wheeler.

October, 1938.



[*Photo: Elliott & Fry*

R. V. WHEELER (1883-1939)

The publishers regret to record the death, on October 28th, 1939, of Professor R. V. Wheeler, joint author of *The Quality of Coke*. It is fitting that this, his last publication, which was ready for issue at the time of his death, should bear his likeness.

RESEARCH PROGRAMMES

The following programmes, which form the basis of the work of the Midland Coke Research Committee, are reproduced from "Coke for Blast Furnaces," 1930. Those starred have been undertaken by the Committee, or by the research organizations with which the Committee is in close touch, namely, the Safety in Mines Research Board and the South Yorkshire Survey Committee of the Fuel Research Board.

Group I. The Constitution and Properties of Coal

Programme No.			
*C.C.	1.	Proximate analysis . .	Volatile Matter.
C.C.	2.	,, ,, . .	Ash.
*C.C.	3.	Ultimate analysis . .	Carbon and hydrogen.
*C.C.	4.	,, ,, . .	Nitrogen.
*C.C.	5.	,, ,, . .	Sulphur.
*C.C.	6.	,, ,, . .	Oxygen.
*C.C.	7.	,, ,, . .	Phosphorus and arsenic.
*C.C.	8.	Rational analysis . .	Hydrocarbons.
*C.C.	9.	,, ,, . .	Resins.
*C.C.	10.	,, ,, . .	Plant remains.
*C.C.	11.	,, ,, . .	Ulmins.
*C.C.	12.	,, ,, . .	The reactivity of the ulmins.
C.C.	13.	,, ,, . .	Sulphur compounds.
*C.C.	14.	,, ,, . .	Nitrogen compounds.
*C.C.	15.	Physico-chemical properties	The "wettability" of coal by resins and hydrocarbons.
*C.C.	16.	,, ,, ,,	The effect of oxidation.
*C.C.	17.	,, ,, ,,	The effect of preheating.

Group II. The Thermal Treatment of Coal

*T.T.	1.	Thermal treatment . .	Distillation in a vacuum.
*T.T.	2.	,, ,, . .	Variation in rate of heating.
*T.T.	3.	,, ,, . .	The determination of the plastic range.
*T.T.	4.	,, ,, . .	The determination of the softening plant.

PROGRAMME NO.		
*T.T. 5.	Thermal treatment	The determination of (*a*) the expansion range (*b*) the amount and force of expansion.
*T.T.		The determination of caking power.
*TT. 7.		Correlation of caking-power with coking properties.
*T.T.		The effect of preheating.
T.T. 9.		The formation of carbon.
*T.T. 10.		Correlation of yields of gas and tar with ultimate and rational analysis.
*T.T. 11.		Exothermic and endothermic reactions during thermal treatment.
T.T. 12.		The effect of mineral matter on the yields of by-products.

Group III. The Production of Commercial Cokes

(*a*) *Coke Production*

*C.P. (*a*) 1.	Coke Production	The effect of oven width.
*C.P. (*a*) 2.	,, ,,	The effect of flue temperature.
*C.P. (*a*) 3.		The effect of methods of charging.
*C.P. (*a*) 4.		The effect of size of coal.
*C.P. (*a*) 5.		The effect of moisture in the coal.
*C.P. (*a*) 6.		The effect of ash (and of free dirt) in the coal.
*C.P. (*a*)		The effect of prolonging heating beyond the burning time.
*C.P. (*a*) 8.		The effect of blending normal coking coal with: (*a*) coke breeze (both from low and high temperature carbonisation); (*b*) anthracite; (*c*) low-oxygen "non-coking" coal; (*d*) high-oxygen "non-coking" coal; (*e*) oxidised coals; (*f*) preheated coals.
*C.P. (*a*) 9.		The effect of blending different coking coals; (*a*) from the same coal field; (*b*) from different coal fields.

(*b*) *Coke Properties*

PROGRAMME NO.		
*C.P. (*b*) 1.	Proximate analysis	Moisture.
*C.P. (*b*) 2.		Volatile matter.
*C.P. (*b*) 3.		Ash.
*C.P. (*b*) 4.	Ultimate analysis	Carbon and hydrogen.
*C.P. (*b*) 5.		Nitrogen.
*C.P. (*b*) 6.		Sulphur.
*C.P. (*b*) 7.		Phosphorus and arsenic.
C.P. (*b*) 8.	Rational analysis	Character of ash.
C.P. (*b*) 9.		Sulphur compounds.
C.P. (*b*) 10.		Phosphorus compounds.
C.P. (*b*) 11.		Carbon compounds and forms of carbon.
*C.P. (*b*) 12.	Physical Properties	(*a*) Bulk density; (*b*) apparent specific gravity; (*c*) real specific gravity; (*d*) percentage total porosity; (*e*) percentage apparent porosity.
*C.P. (*b*) 13.		Macroscopic examination.
*C.P. (*b*) 14.		Microscopic examination.
*C.P. (*b*) 15.		Mechanical strength. Shatter test. (Impact hardness).
*C.P. (*b*) 16.		Mechanical strength. Abrasion test. (Surface hardness.)
C.P. (*b*) 17.		Mechanical strength. Resistance to compression.
*C.P. (*b*) 18.	Combustion	Rate of combustion. Combustion temperature.
C.P. (*b*) 19.		Reactivity towards carbon dioxide.
C.P. (*b*) 20.		Reactivity towards steam.
C.P. (*b*) 21.		Ignition temperature.
*C.P. (*b*) 22.		Clinker formation.

CONTENTS

PART I

INTRODUCTION

CHAPTER I

PAGE

STANDARDS FOR COMMERCIAL COKE 3

Coke for grates, stoves and boilers—Coke for hearths and braziers—Coke for metal melting—Coke for cupolas—Coke for gas-producers—Coke for blast-furnaces—The most useful qualities of a coke: purity; size; hardness; combustibility and specific reactivity.

CHAPTER II

STAGES IN THE FORMATION OF COKE 11

Pre-softening—Decomposition and softening—Swelling and agglomeration—Solidification and hardening.

CHAPTER III

THE CHARACTERISTICS OF COALS WHICH INFLUENCE THE FORMATION OF COKE FROM THEM 15

Ultimate analysis—The blending of coals.

CHAPTER IV

THE INFLUENCE OF CARBONISING CONDITIONS ON THE FORMATION OF COKE 17

The size of the coal—The bulk density of the charge—The rate of heating.

PART II

THE STANDARDS OF A COMMERCIAL COKE

CHAPTER V

THE SIZE OF COKE 23

The size of coke from the ovens: the effect of the quality of the coal; the effect of the size of the coal; the effect of blending; the effect of the method of charging the oven; the effect of the rate of heating—The size of coke at the blast-furnace: the effect of handling—The size of coke for cupolas—The size of coke for stoves and boilers.

CHAPTER VI

THE STRUCTURE OF COKE 41

CHAPTER VII

PAGE

THE SAMPLING AND ANALYSIS OF COKE 42

The sampling of coke—An experimental study of the sampling of oven coke for moisture determination: the collection of a gross sample of coke; reduction of the gross sample for analysis; sampling ½-in. coke for determination of moisture—Sampling coke for the determination of ash—The influence of the weather on the moisture-content of coke—Sampling for the shatter test—The analysis of coke: moisture; ash; volatile matter; nitrogen; carbon—Appendix: tests on the moisture contents of cokes quenched in different ways.

CHAPTER VIII

THE DENSITY AND POROSITY OF COKE 70

Apparent density: displacement and boiling methods; the cage test—Porosity—Bulk density—Real density: the effect of differences of temperature during a determination; the effect of the size of the coke; the effect of incomplete wetting; the effect of adsorption—The significance of the real density of coke—Appendix I: the determination of the density and porosity of small specimens of coke—Appendix II.

CHAPTER IX

THE ABRADABILITY OF COKE 96

The Cochrane test—The effect of the shale-content of coke on its abradability—The effect of the structure of coke on its abradability—The effect of blending coking slacks with coke dust on the abradability of coke—Accuracy of the Cochrane test—Relationship between Cochrane index and shatter test index—The Sheffield coke abrasion test—Appendix: specification for the drum abrasion test.

CHAPTER X

THE HARDNESS OF COKE 115

The shatter test—The accuracy of the shatter index: true shatter index; average error; probable error; errors having a greater than even chance; the number of tests required to ensure a specified degree of accuracy—The influence of the hardness of coke on blast furnace performance—Sampling of coke for the shatter test—Appendix, solutions of the probability integral.

CHAPTER XI

THE REACTIVITY OF COKE 132

The influence of the size of the coke—Reactions within a burning bed of coke: reactions in the hearth of a blast furnace; reactions in a cupola; the combustion of coke in steam boilers; the combustion of coke in central-heating and domestic boilers; the combustion of coke in open grates—Appendix I: the determination of the "critical air blast"—Appendix II: test coke grate and water flow radiometer—Appendix III: the wet oxidation of coke—Appendix IV: fast driving coke for blast-furnaces.

CHAPTER XII

THE SPECIFICATION OF COKE 175

Introduction—The rate of drive of blast furnaces—The average quality of Midland cokes: moisture content; ash content; hardness—Future demands for coke—The value of coke—The specification of coke; sampling; analysis; bonuses and penalties; final assessment of coke quality; detailed methods for sampling and analysis at the coke ovens and blast furnaces.

PART III

THE FORMATION OF COKE

CHAPTER XIII

PAGE

THE COMPOSITION OF COAL 199

The coal band—The classification of coals—The relationships between ultimate analysis and volatile matter and calorific value—Coking coals: swelling power; the relationship between ultimate analysis and yields of by-products; tar; thermal yields of gas—Banded bituminous coals; durain and clarain; vitrain; fusain—The microstructure of banded bituminous coals—Appendix: the recovery of fusain; the estimation of fusain, the determination of the durain content of a coking slack.

CHAPTER XIV

THE DECOMPOSITION OF COAL 231

The decomposition point of coal: the decomposition of coal by slow distillation; the decomposition of coal at a rate of heating of 1° C. per min.—Coke formation and the release of volatile matter—The evolution of volatile matter from coal at constant volume—The quality of the "volatile matter" evolved from coal at different temperatures—The rate of production of gas from different coals.

CHAPTER XV

THE SOFTENING AND SWELLING OF COAL 271

The softening-point of coal—The initial expansion of columns of particles.

CHAPTER XVI

THE PLASTICITY OF COAL AND THE FORMATION OF SEMI-COKE . 294

The determination of the duration of plasticity of coal free to expand—The influence of the external pressure on the plasticity of coal—Methods of measuring the plasticity of coal—The relationship between the end of plasticity, evolution of volatile matter and the porosity of cokes.

CHAPTER XVII

THE FORMATION OF COKE FROM SEMI-COKE 309

The loss of volatile matter—Changes in reactivity—Formation of graphite.

CHAPTER XVIII

THE MECHANISM OF COKE FORMATION . . 326

The "coking constituents" of coal—The importance of the insoluble portion of coal—The adsorption of oil during the formation of coke—Surface-flow during the formation of coke—The effect of addition of various substances to coal before carbonisation—The inimical effect of gases and vapours produced during plasticity.

CHAPTER XIX

PAGE

THE MEASUREMENT OF THE SWELLING POWER OF COAL . . 343

Sheffield laboratory coking test—Specification and method of testing—The correlation of swelling power with the ultimate analysis of coal—The determination of the swelling power of lump coal—The swelling power of coal at a rapid rate of heating: B.S.I. crucible swelling test; method of testing and reporting—Relationship between B.S.I. swelling power and composition of coal—The measurement of caking (agglutinating) power.

PART IV

IMPROVING THE QUALITY OF COKE

CHAPTER XX

THE INFLUENCE OF THE SIZE AND PURITY OF THE COAL . . 373

The effect of size—The effect of shale content.

CHAPTER XXI

THE INFLUENCE OF THE METHOD OF CHARGING AN OVEN . 384

CHAPTER XXII

THE INFLUENCE OF THE RATE OF HEATING AN OVEN . . 389

CHAPTER XXIII

THE INFLUENCE OF BLENDING THE COAL CHARGE . . . 405

The blending of two coking coals—The blending of a non-coking coal with a coking coal—Blending with durain and fusain—Blending with coke dust—An attempt to find a substitute for fusain—A comparison of the use of fusain and coke dust—General conclusions.

NAME INDEX 459

SUBJECT INDEX 461

LIST OF ILLUSTRATIONS

W. J. Brooke, Esq., Chairman, Midland Coke Research Committee *Frontispiece*

PART II.—THE STANDARDS OF A COMMERCIAL COKE

FIG.		PAGE
1.	The Effect of Screening out Breeze of different Maximum Size on the Operation of a Blast Furnace. Average Monthly Data	25
2.	The Effect on the Screen Size of Coke of Blending Parkgate M. with Barnsley B. Coal in Different Proportions	28
3.	The Effect on the Screen Size of Coke of Adding Fusain to a Blend of 50 per cent. Parkgate M., 50 per cent. Barnsley B.	28
4.	The Effect on the Screen Size of Coke of Adding Strongly-coking Coals to a Derbyshire Coking Slack	29
5.	The Effect on the Screen Size of Cokes of Varying the Coking Time in an 18-in. Oven. Three Coals	30
6.	The Effect, on the Screen Size of Coke, of each Drop during Handling between the Coke Ovens and Blast Furnace	33
7.	The Increase in Impact Hardness of Coke on Handling shown by Decrease in Percentage Undersize Produced in Successive 6-ft. Drops	35
8.	The Increase in Impact Hardness on Handling is Proportional to the Original Shatter Index of Midland Cokes of Average Hardness	35
9.	Comparison of Large (above) and Small-Pored (below) Cokes of the Same Total Porosity	39
10.	The "Average Deviations" in Sampling Cokes of Different Moisture Contents	45
11.	Diagrammatic Representation of Jaw Granulator for Crushing Coke to ½-in. Size	51
12.	Six-inch Manganese-Steel Mortar and Long-handled Pestle for Pulverising Coke	60
13.	Average Error of the Mean Apparent Specific Gravity for Various Groups of Tests	71
14.	Diagrammatic Representation of the Cage Test for Apparent Specific Gravity	73
15.	The Relationship between Bulk Density and Porosity of a Coke (Killing)	80
16. 17.	Apparatus for the Determination of the Real Specific Gravity of Coke	85
18.	Chart for Correcting Real Specific Gravity for Changes of Temperature during a Determination	92
19.	The Effect of Ash Content on the Cochrane Index of Two Cokes	97

FIG. PAGE
20. "Pebbly" Structure of Coke due to Non-Coking Constituent 100
21. Irregular Structure of Gas Cokes and Coalite compared with Uniform Structure of Oven Coke (×1/3) . . . 101
22. Errors in the Means of Groups of Tests, Cochrane Index and ½-in. Shatter Index 106
23. Comparison of Mean ½-in. Shatter Index and Mean ⅛-in. Cochrane Index. Values for Different Periods . . 107
24. Comparison of Mean Values of ½-in. Shatter Index and ⅛-in. Cochrane Index for Period of Seven Months . . . 108
25. Percentages of Different Sizes of 4 Cokes remaining after Tests in Sheffield Abrasion Drum with Different Percentage Fillings 109
26. The Relationship between ½-in. Shatter Index and Sheffield Abrasion Index 111
27. The Drum Abrasion Test Apparatus. (*a*) Vertical Section through Drum; (*b*) Charging-hole Cover . . . 113
28. The Chance of Occurence of Different Errors in the Mean 1½-in. Shatter Index. Yorks. III Coke with Various Groups of Tests Averaged 123
29. Probability 9 : 1. Errors which may occur once in 10 times in the Averages of Various Groups of Tests. Cokes of Different Average Error in 1½-in. Shatter Index . . 126
30. Probability 99 : 1. Errors which may occur once in 100 times in the Average of Various Groups of Tests. Cokes of Different Average Error in 1½-in. Shatter Index . . 127
31. Enlarged Sections through Partially-burnt Coke, one Face being Infilled with Plaster-of-Paris. (×4) . . . 135
32. The Effect of Size of Coke on the Reactions in a Fuel Bed. The Relationship between the Surface Area of Coke and the Disappearance of Oxygen 139
33. "Combustibility" Furnace, 24-in. Diameter . . . 142
34. Carbon Dioxide at Different Levels of a Fuel Bed with Different Sizes of Coke (No. 79, average for two analyses) . 146
35. The Temperatures at Different Levels of a Fuel Bed with Different Sizes of Coke (No. 79) 146
36. } The Difference in Character of Clinker on Grate Bars when
37. } Burning Large and Small Coke under the same Conditions 147
38. Comparison of the Weight and Surface Area of Different Sizes of Coke for the Combustion of 1-lb. per sq. ft. per hr. 150
39. The Effect of Rate of Combustion on the Maximum Combustion Temperature in a Fuel Bed of Coke of Standard Size 152
40. "Combustibility" Furnace, 8-in. Diameter . . . 153
41. The Effect of Rate of Combustion and Size of Furnace on the Reactions in a Fuel Bed 155
42. The Effect of Method of Air Supply on the Radiation from Different Sizes of Low Temperature Coke burnt in an Open Grate 160
43. The Relationship between the C.A.B. of Coke and the Time for "Bright Fire" in an Open Grate 161
44. The Effect of Size on the Time for "Bright Fire" for Cokes of Different C.A.B. 164

45. The Effect of Size of Coke on the Mean Hourly Radiation per lb. of Fixed Carbon 164
46. The Effect of Size of Coke on the Maximum Radiation in an Open Grate (Cokes listed in Fig. 44) 165
47. The Effect of Size of Coke on the Mean Temperature of the Surface of Fires in an Open Grate 165
48. Apparatus for Determining Critical Air Blast (C.A.B.) Northern Coke Research Committee 168
49. Accelerated Combustion of Fuels at the Critical Air Blast (N.C.R.C.) 169
50. Apparatus for Determining Critical Air Blast. Midland Coke Research Committee 168
51. Arrangement for Determining Radiation from a Coke Fire. Eagle Grate No. 214 and Radiometer 171
52. View of Radiometer 173
53. Apparatus for Determining the Wet Oxidation of Coke (N.C.R.C.) 172
54. Average Pig Iron Production per Furnace for Great Britain, 1875–1937 177
55. Graph Illustrating Deterioration of Quality of Coke (with increased Consumption per ton of Pig) in Periods of Great Demand (and High Prices) 179
56. Mean Annual Moisture Content of Yorkshire Cokes for Periods of Years Compared with Rainfall and Total Coke Production 181
57. Comparison of Mean Quarterly Moisture-Contents of Cokes Centrally-Quenched and Hand-Quenched. Comparison of Mean Quarterly Moisture-Contents of Yorkshire Cokes and Mean Rainfall 183
58. The Relationship between Mean Quarterly Moisture Content, Rainfall and Evaporation 184
59. Trends of Production of Steel in the World and in Great Britain over Period 1870–1940 187

PART III.—THE FORMATION OF COKE

60. The Relationship between Carbon and Oxygen Contents of Coals, Lignites to Anthracites (Hickling) 200
61. The Relationship between Carbon and Hydrogen Contents of Coals, Lignites to Anthracites (Needham) 201
62. The Relationship Carbon to Hydrogen for Typical British Coals 203
63. The Relationship Volatile Matter to Calorific Value for Typical British Coals (Cf. Fig. 62) 203
64. The Variation of Carbon and Hydrogen in Different Sections of Seams in Yorkshire (above) and Durham and Northumberland (below) 204
65. The Relationship between Swelling-power and Carbon and Hydrogen Contents 207
66. Lines of Equal Tar Yields for Canadian Coals of Different Carbon and Hydrogen Contents. Experimental Coke Oven 208
67. The Relationship between Thermal Yields of Gas and Carbon and Hydrogen Contents of Coal 208

FIG. PAGE
68. The Relationship between Thermal Yields of Gas and Volatile Matter and Calorific Value of Coal . . . 210
69. The Relationship between Thermal Yields of Gas, Swelling Power and Carbon and Hydrogen Contents of Coal . . 211
70. Photograph of Grey and Black Durain in Upper Beeston Coal (Wandless and Macrea) 217
71. The Effect on the Swelling Power of Bright Coal of Adding Varying Amounts of Durain from the Same Seam . . 221
72. The Effect of Blending Bright Coal and Durain in Different Proportions on the Appearance of Coke made in a Capsule Test. (Figures are per cent. of Bright Coal.) . . . 223
73. The Comparative Effect on the Swelling Power of Bright Coal of Adding Durain or Fusain 226
74. Comparison of the Appearance of Fusain and Bright Coal Under Different Conditions 227
75. The Reactivity to Permanganate of Coals of Varying Carbon Content. (Francis) 231
76. Permanganate Numbers of Coals Heated to Different Temperatures 233
77. Graph illustrating Method of Determining the Decomposition Temperature of Coal (Hibbott and Wheeler) . . . 237
78. Evolution of Gas, Adsorption and Distillation of Oil from Coals of 89 to 84 per cent. Carbon Content on Heating to Different Temperatures (T° C.) at a Rate of 1° C. per min. The Effect of Residual Oil on the Shatter Index of Coke made on Heating Coal Residues at T° to 600° C. . 240
79. Evolution of Gas, Adsorption and Distillation of Oil from Coals of 83 to 77 per cent. Carbon Content on Heating to Different Temperatures (T° C.) at a Rate of 1° C. per min. Effect of Residual Oil on Shatter Index of Coke made on Heating Residues at T° C. to 600° C. 241
80. Per cent. of Total Oil distilled from Columns of Particles of Coals of Different Carbon Content at Various Temperatures 243
81. Total Evolution of Gas from Columns of Particles of Coals of Different Carbon Content at Various Temperatures . 243
82. Comparison of Evolution of Oil from Columns of Particles and from Single Particles of Different Carbon Content . 245
83. Apparatus for Studying the Evolution of Gas from Coal (*a*) Free to Swell (*b*) Prevented from Swelling . . . 252
84. Evolution of Gas from Coals (*a*) Free to Swell (*b*) Prevented from Swelling 253
85. Evolution of Gas and Tar from Coal (*a*) Free to Swell (*b*) Prevented from Swelling 254
86. Apparatus for Studying the Quality of Coke made from Coal Prevented from Swelling 255
87. Davidson's Modification of Foxwell's Apparatus for Determining the Plastic Range of Coal 256
88. The Quality of the Volatile Matter from Coals of Different Carbon Content, and at Different Temperatures . . 257
89. Total Yields of Gas from Coals of Different Carbon Content over the Range 350–430° C. at a Rate of Heating of 1° C. per min. 264

90. Total Yields of Gas from Coals of Different Carbon Content over the Range 380–600° C. at a Rate of Heating of 1° C. per min. 265
91. Total Yields of Gas to 900° C. at Different Rates of Heating (Kent Thick Coal) 267
92. Silica Apparatus for Sheffield Laboratory Coking Test . 272
93. Appearance of Residues from Columns of Particles Heated to Various Temperatures in the Sheffield Laboratory Coking Test. R.M. Parkgate Coal. (The Coalescence Index is the percentage remaining on 60 mesh after dropping 3 times through a height of 3-ft.) 270
94. Rose Sections of 1-in. Cubes of Coal Heated to Different Temperatures. R.M. Parkgate Coal (×1) (See Fig. 95 for enlargements of rectangles marked) 274
95. Rose Sections of 1-in. Cubes of Coal Heated to Different Temperatures. R.M. Parkgate Coal (×10) (see also Fig. 94) 275
96. Comparison of (*a*) Changes in Volume, (*b*) Evolution of Gas and Oil from Single Lumps and Columns of Particles at Different Temperatures. R.M. Parkgate Coal. . . 278
97. Softening Points of Coals in the Sheffield Laboratory Coking Test, Compared with Carbon Contents (Dry, Ash-free) . 280
98. Rose Sections of 1-in. Cubes of Coal Heated to 380° C., Pressed and not Pressed. R.M. Parkgate Coal . . 282
99. Apparatus used to Measure the Effect of Different Loads on the Swelling of Coal 283
100. The Effect of Pressure on the Softening Temperatures of Various Coals 284
101. The Increased Reactivity of Coals at their Softening Temperatures (S.T.) 286
(With some Coals, other Critical Points occur at the Temperatures of Initial Expansion (I.E.) and of Final Expansion (F.E.))
102. The Formation of Closed Pores at the Temperature of Initial Expansion. R.M. Parkgate Coal (×10) (see also Fig. 103) 288
103. The Formation of Closed Pores at the Temperature of Initial Expansion. R.M. Parkgate Coal (×100). Series I—Under no load. Temperature of Initial Expansion 401° C. (cf. Fig. 102) 289
104. Diagrammatic Representation of the Neutralisation of the Vertical Components of Swelling Pressure in a Coke Oven 298
105. The Load required to Prevent Swelling and to Cause the Plunger to begin to Sink through the Charge. Various Coals 300
106. Increase in Surface Hardness of Cokes from 5 Coals of Different Carbon Content (C. %) on Carbonising at Temperatures of 500 to 1100° C. Mean Specific Gravities and Mean Volatile Contents of the Cokes 310
107. The Mean Changes in Composition of Cokes between 500 and 1100° C. made from Coals of Table CXVI . . . 313
108. Individual Changes in Carbon Contents of Cokes between 500 and 1100° C. made from Coals of Table CXVI . . 315

FIG. PAGE
109. Individual Changes in Oxygen Contents of Cokes between 500 and 1100° C. made from Coals of Table CXVI . . 315
110. Changes in the Properties of Cokes made at Different Temperatures (S. Met. Gas Co.) 317
111. The Relationship between the Electrical Resistance of Cokes made at Different Temperatures and their Volatile Matter Contents (Davis and Auvil) 320
112. The Wet Oxidation Indices of Cokes made at Different Temperatures (Blayden and Riley) 322
113. Microphotograph (×140) of unheated Briquette of Coal Polished with Rouge, Showing Outlines of Original Particles 334
114. Microphotographs (×140) of Briquette of Coal Heated to Different Temperatures and Polished with Rouge. Briquettes show Surface Flow at a Temperature of 445° C. . 336
115. The Sheffield Laboratory Coking Test Using Steel Retort and Plunger 344
116. Difference in Temperature between Coal and Furnace in Sheffield Laboratory Coking Test 345
117. The Height of the Coal Charge in the Sheffield Laboratory Coking Test after Dropping the Plunger from Different Heights 346
118. The Relationship between the Composition of Coal and its Swelling Power in the Sheffield Laboratory Coking Test. Iso-hydrogen Lines 353
119. The Relationship between the Composition of Coal and its Swelling Power in the Sheffield Laboratory Coking Test. Iso-carbon Lines 357
120. The Relationship between the Composition of Coal and its Swelling Power in the Sheffield Laboratory Coking Test. Iso-swelling Lines 357
121. Volumenometer for Determining the Swelling Power of Lump Coal (Scale one-quarter full size) 359
122. The Apparatus for the B.S.I. Crucible Swelling Test . . 362
123. The Standard Profiles of Coke Buttons and Swelling Numbers, B.S.I. Crucible Swelling Test 363
124. Apparatus for Viewing the Button to avoid Parallax, B.S.I. Crucible Swelling Test 364
125. The Relationship between the B.S.I. Crucible Swelling Number and the Carbon and Hydrogen Contents of Coals. Iso-hydrogen Lines 365
126. The B.S.I. Crucible Swelling Numbers Superimposed on Seyler's Classification of Coal 366
127. The Influence on the B.S.I. Crucible Swelling Number of Blending Durain with Bright Coal 367

PART IV.—IMPROVING THE QUALITY OF COKE

128. Longitudinal Section of Durham Coke made from Coal Cleaned by Froth Flotation 375
129. Rose Sections of "Capsule" Cokes made from Parkgate Durain of Different Sizes (×1) 379

FIG. PAGE

130. Microphotograph (×10) of "Capsule" Coke made from Parkgate Durain of ⅛--1/10-in. size to Show Agglomeration of Different Particles (Cf. Fig. 129) 379
131. Longitudinal Sections of Cokes made from Halifax Soft Coal of Different Ash Contents (*a*) From Coal of 10·0% Ash (*b*) From Coal of 5·5% Ash. Note Particles of Shale in (*a*) with Numerous Small Fractures 381
132. Sectional Drawings of Experimental Oven and Burner . 390
133. View of the Experimental Oven 391
134. Another View of the Experimental Oven with Door Shield, in the Shed. Shatter Test Apparatus 395
135. The Effect of Varying the Coking Time on the 1½-in. Shatter Index and the Percentage of over 4-in. Coke. Parkgate and Silkstone Slack 397
136. The Effect of Varying the Coking Time on the 1½-in. Shatter Index and the Percentage of over 4-in. Coke. Parkgate R. Slack 398
137. The Effect of Varying the Coking Time on the 2-in. Shatter Index and the Percentage of over 2-in. Coke. Barnsley B.M. Slack 399
138. The Effect of Varying the Coking Time on the Shatter Indices of the Cokes. West Yorkshire G. Slack. 401
139. Comparison of the Effect of Varying the Coking Time on the 1½-in. Shatter Index of Coke made in Experimental Ovens of 18-in. and 16-in. width 403
140. The Effect of Blending Parkgate C. Slack with Silkstone C. Slack in Different Proportions. The Addition of 4 per cent. Fusain to one Blend. Shatter Indices of Cokes . 406
141. The Effect of Blending Parkgate C. Slack with Halifax Soft Slack in Different Proportions. Shatter Indices of Cokes 408
142. The Effect of Blending Parkgate C. Slack with West Yorkshire G. Slack in Different Proportions. Shatter Indices of Cokes 410
143. The Effect of Blending Four Strongly Swelling Coals with Derbyshire Slack in Different Proportions. Shatter Indices of Cokes 412
144. The Effect of Blending Barnsley B.M. Slack with Parkgate M. Slack in Different Proportions. The Effect of Adding Fusain to Several Blends. Shatter Indices of Cokes . 417
145. The Effect of Blending Barnsley B.M. Slack with Silkstone C. Slack in Different Proportions. The Effect of Adding Varying Amounts of Fusain to one Blend. Shatter Indices of Cokes 419
146. The effect of Blending Barnsley K. Slack with Parkgate R. Slack in Different Proportions. The Effect of Storage. Shatter Indices of Cokes 420
147. The Effect of Blending Barnsley R. Slack with Parkgate-Barnsley D. Slack in Different Proportions. Shatter Indices of Cokes 422

FIG. PAGE

148. The Effect of Blending Barnsley H.M. Slack with Parkgate and Silkstone Slack and Barnsley S.K. Slack in Different Proportions. The Effect of Size. Shatter Indices of Cokes 424

149. The Effect of Adding Durain and Fusain to Barnsley H.M. Bright Coal. Shatter Indices of Cokes 429

150. The Effect of Durain Content on the Shatter Indices of Cokes. Variation of Natural Durain Content and Added Durain 432

151. The Effect of Adding Varying Amounts of Fusain to Parkgate and Silkstone Nut Coal. Shatter Indices of Cokes . 435

152. The Effect of Progressive Addition of Coke Dust to Parkgate and Silkstone Slack. Shatter Indices and Percentage of over 4-in. Coke 445

153. The Effect of Adding Coke Dust to Parkgate and Silkstone Coals of Varying Durain and Fusain Contents . . 449

154. Comparative Grindability of Cokes and Semi-Cokes made at Different Temperatures 451

155. Relationship between the Carbon Contents of Single Coals and the Shatter Indices of their Cokes (Evans and Ridgion) 456

156. The Relationship between the Carbon Content of "40 per cent." Blends and the Shatter Indices of their Cokes, expressed on a Background of the Band taken from Fig. 155 457

LIST OF TABLES

TABLE NO.		PAGE
I.	The Effect on Coke Consumption in a Blast-furnace of removing Different Quantities of Breeze	24
II.	The Effect of the Quality of the Coal on the Size of Coke. (Tests in an Experimental Oven)	26
III.	The Effect of Fine Grinding of Coal on the Size of Coke	27
IV.	The Handling of Coke	31
V.	Details of Handling between Coke-ovens and Blast-Furnaces	32
VI.	Size of Coke at Different Points of System in Table V	33
VII.	Comparison of Ungraded and Graded Coke in a Cupola	36
VIII.	Summary of Tests on Sampling Coke Quenched by Water-spray	44
IX.	Summary of Tests on Sampling of Coke Quenched Centrally	46
X.	Summary of Tests on Sampling of Coke Quenched by Hand-Hose	47
XI.	Minimum Weights of Gross Sample	49
XII.	Average Error in Moisture-Tests, obtained by Sampling Wagons	49
XIII.	The Sampling of Coke for the Determination of Ash	54
XIV.	The Sampling of Coke for the Determination of Ash	55
XV.	The Effect of Size of Coke on Ash-content	56
XVI.	Loss of Moisture from Coke in Air, per cent.	57
XVII.	The Effect on the Ash-content of Coke of the Method of Preparing the Sample	60
XVIII.	Test No. I, Variation of Moisture content of 48 Increments of 1 lb. (Two passes under spray)	62
XIX.	Test No. II. Variation of Moisture content of 48 Increments each of 5 lb., 2½ lb., and 1 lb. (Three passes under spray)	63
XX.	Test No. III. Variation of Moisture content of 48 Increments each of 5 lb., 2½ lb. and 1 lb. (Four passes under spray: 2 fast, 2 slow)	64
XXI.	Test No. IV. Variation of Moisture content of 48 Increments each of 5 lb., 2½ lb., and 1 lb. (Four passes under spray)	65
XXII.	Test No. V. Variation of Moisture content of 48 Increments each of 5 lb., 2½ lb. and 1 lb. (Six passes under spray: 4 fast, 2 slow)	66
XXIII.	Variation of Moisture content of 48 Increments each of 5 lb., 2½ lb. and 1 lb. (Central Quenching, 4,000 gallons of water discharged in 35 sec. on to 8 tons of coke)	67

TABLE NO.
XXIV. Variation of Moisture content of 48 Increments, each of 5 lb., 2½ lb. and 1 lb. (Quenched with Hand Hose on Inclined Bench) 68
XXV. Variation of Moisture content of 48 Increments of 5 lb., 2½ lb. and 1 lb. (Second Test at Plant using Quenching by Hand Hose on an Inclined Bench) . 69
XXVI. The Apparent Density of Coke 70
XXVII. Apparent Densities. Percentages of Cokes having Different Average Deviations from Mean Values . 72
XXVIII. Apparent Densities. "Cage" Tests on Three Cokes . 74
XXIX. Apparent Densities of Parkgate Coke. The Effect of Size of the Coke 74
XXX. Apparent Densities. "Cage" Tests on Derbyshire Coke 75
XXXI. Comparison of Apparent Densities by "Boiling" and "Cage" Methods. (Northern Coke Research Committee) 76
XXXII. Effect of Rate of Heating on the Porosity of Coke . 77
XXXIII. Mean Porosity of Cokes from Top and Cake Charges in Different Districts 78
XXXIV. The Porosity of Coke and its Permeability to Gases . 79
XXXV. Relationship between Porosity and Bulk-Density of Coke. (Killing) 80
XXXVI. Error $\left[\frac{2(X-Y)}{X-Y+d_{16}}\right]$ in Real Specific Gravity of Coke for 1° C. Increase in Temperature during a Test using Different Liquids in a Specific Gravity Bottle 83
XXXVII. Properties of Liquids in Specific Gravity Determinations 84
XXXVIII. The Effect of Size of Coke on the Value for Real Specific Gravity 86
XXXIX. The Effect of Air in Causing Incomplete Wetting . 87
XL. High Values with Method III. Coke No. 30 B (Durham) 88
XLI. Further Tests on the Formation of an Adsorbed Film 89
XLII. Mean Values of Six Determinations by Various Methods 90
XLIII. The Use of Organic Liquids in Determining the Specific Gravity of Coke 90
XLIV. The Rank of Coal and the Real Density of its Coke . 93
XLV. The Abradability of South Yorkshire Cokes. Cochrane's Test 98
XLVI. Effect of Ash-content of Coke No. 49 on Cochrane Abrasion Index 99
XLVII. Repetition Tests on Coke 104
XLVIII. Average Errors of Shatter Indices, Yorkshire Cokes . 119
XLIX. The Average Errors of Groups of Shatter Tests. . 120
L. Average and Probable Errors of 1½-in. Shatter Indices of Cokes 121
LI. Errors of 9 to 1 Probability in 1½-in. Shatter Index. Single Tests and Groups of Tests 124

TABLE NO.		PAGE
LII.	Errors of 99 to 1 Probability in 1½-in. Shatter Index. Single Tests and Groups of Tests	124
LIII.	The Number of Shatter Tests Required to Ensure a Specified Degree of Accuracy	125
LIV.	The Average Errors for the 1½-in. Shatter Index of Midland Cokes	128
LV.	Relative Reactivities of Cokes and Coals	132
LVI.	Relative Reactivities of Cokes Made at Different Temperatures (Permanganate Numbers)	133
LVII.	Surface-Areas of One Cu. Ft. of Different Sizes of Coke	134
LVIII.	Porosity of Pieces of Coke at Different Levels of a Furnace (Coke No. 31C)	137
LIX.	Increase in Porosity on Burning Retort Carbon	138
LX.	Properties of Cokes Used for Combustion Tests	143
LXI.	Effect of Size of Coke on Rate of Supply of Air	143
LXII.	Combustion of Cokes of Different Sizes. Two-ft. Diameter Furnace	144
LXIII.	Central-Heating Boilers. Dimensions and Rating	157
LXIV.	Mean Rates of Combustion of Cokes in Open Grates	162
LXV.	The Mean Ash-contents of Yorkshire Cokes, 1931–1937	185
LXVI.	The Mean 1½-in. Shatter Indices of Yorkshire Cokes, 1931–1937	185
LXVII.	Estimates of Requirements of Coke in the Midlands	186
LXVIII.	Grüner's Classification of Coals	201
LXIX.	A.S.T.M. Classification of Coals According to Rank	202
LXX.	Analyses of Typical English Coals *facing p.*	204
LXXI.	The Relationship between the Calorific Value and the Carbon and Hydrogen Contents of Coals. (Parr's "Unit Coal" Basis)	206
LXXII.	The Relationship between the Volatile Matter and the Hydrogen and Carbon Contents of Coals. (Parr's "Unit Coal" Basis)	206
LXXIII.	The Relationship between the Analysis of Coals and the Quality of Their Cokes	212
LXXIV.	The Proportions of Durain and Fusain in Coal Seams	214
LXXV.	The Amounts of Durain in Small Coal from the Barnsley Seam	214
LXXVI.	Amounts of Durain in Graded Sizes of (Mixed) Parkgate and Silkstone Seams	215
LXXVII.	Analyses of Clarains and Durains of Yorkshire Coal Seams	216
LXXVIII.	Analyses of Clarains and Durains of Seams in Different Coalfields	216
LXXIX.	Analyses of Grey and Black Durain in Upper Beeston Seam	219
LXXX.	Analyses of Vitrain and Clarain in Yorkshire Coking Coals	220
LXXXI.	Analyses of Fusain and Bright Coal	222
LXXXII.	Analyses of Slacks from the Barnsley Seam and of the Durain and Bright Coal in Them	225

TABLE NO.		PAGE
LXXXIII.	The Reactivity of Heated Coals. Analytical and Test Data for the Coals Used	232
LXXXIV.	The Decomposition-Points of Coals (Holroyd and Wheeler)	235
LXXXV.	Changes in Composition of Gases and Oils with Temperature for Barnsley Vitrain (V) and Durain (D)	236
LXXXVI.	The Decomposition-Points of Coals	238
LXXXVII.	Comparison of the Production of Gas from Coal at Different Rates of Heating	242
LXXXVIII.	The Evolution of Oil from Lump and Crushed Coal	244
LXXXIX.	The Loss of Volatile Matter from Coal During Heating (Davidson)	247
XC.	The Loss of Volatile Matter from Coal during Heating (Davidson)	249
XCI.	The Plasticity of Coal and the Evolution of Volatile Matter (Hofmeister)	250
XCII.	The Plasticity of Coal and the Evolution of Volatile Matter. (Bunte, Brückner and Ludewig)	251
XCIII.	The Plastic Range of Coal (Foxwell's Method)	258
XCIV.	The Quality of the Volatile Matter Evolved from Coal	262
XCV.	The Evolution of Volatile Matter from Coal during Different Ranges of Temperature	263
XCVI.	A Comparison of the Swelling-Ranges of Vertical and Horizontal Charges of Coal	263
XCVII.	The Production of Gas from Coal Heated at a Rate of 1° C. per Minute. C.c. per gramme	266
XCVIII.	Evolution of Gases (c.c. per gramme) from Lump and Powdered Coal per 10° C. Rise in Temperature	277
XCIX.	Change in Height of R.M. Parkgate Coal, per cent.	279
C.	Swelling of Lumps of Coal near their Softening-Point	281
CI.	The Expansion of 1-in. Cubes of R.M. Parkgate Coal (per cent.)	281
CII.	The Expansion of Cubes of R.M. Parkgate Coal	282
CIII.	Comparison of Lowest Temperatures of Initial Contraction of Coals (under Pressure) with their Active Decomposition-Points	285
CIV.	The Effect of Pressure on the Temperature of Initial Expansion of a Column of Particles of Coal (R.M. Parkgate)	291
CV.	The Formation of Pores at or near the Temperature of Initial Expansion. (R.M. Parkgate Coal)	292
CVI.	The Determination of the Duration of Plasticity of a Coal (R.M. Parkgate) Free to Expand	295
CVII.	Solidification-Temperatures of Coals Free to Expand	295
CVIII.	Swelling, Per cent., for Successive 5° C. Rises in Temperature above the Temperature of Initial Expansion	295

TABLE NO. PAGE

CIX. Ratios for the Evolution of Gas over Ranges of 10° C. for Different Coals 296
CX. The Effect of Pressure on the Heating of Coal. (Coal used, R.M. Parkgate) 299
CXI. U.S. Bureau of Mines Investigation of Plastic Properties of Coal 302
CXII. Plasticity Measurements of Gieseler 304
CXIII. Plasticity Phenomena of Coal (Pieters, Koopmans and Hovers) 305
CXIV. Percentages of Open and Closed Pores in Cokes Made at Constant Volume 307
CXV. Analyses of Some Coals Examined in the B.M.–A.G.A. Test 309
CXVI. Ultimate Analyses (dry, ash-free) of Cokes made at Different Temperatures 311
CXVII. Comparison of Calculated and Actual Composition of Coked Residues 314
CXVIII. Per Cent. of Weight of Different Gases on Heating Coals to Different Temperatures 316
CXIX. Electrical Conductivity of Coked Residues from Coals of Different Rank 318
CXX. Electrical Resistance of Cokes made at Different Temperatures (in ohms per meter per sq. mm.) . 319
CXXI. The Wet Oxidation of Coke (Blayden and Riley) . 321
CXXII. Wet Oxidation Indices of Cokes from Different Coals Formed at 900° C. 323
CXXIII. Properties of Successive Fractions Floated from Ruhr Coke 324
CXXIV. Per cent. of Flotation Residues from Cokes made from Different Coals 325
CXXV. Effects of Adding Tar to Non-coking Coal before Carbonising 338
CXXVI. Effect of Adding Tar to Poor Coking Coal before Carbonising 338
CXXVII. Effect of Rank of Coal on Retention of Added Tar on Preheating to 420° C. 339
CXXVIII. Effect of Adding 4 per cent. of Different Substances on the Swelling Power of Coal . . 340
CXXIX. Effect of Distilling Coal in a Stream of Carbon Dioxide 340
CXXX. The Effect of Carbonising Coal in Streams of Gases and Vapour 341
CXXXI. Sheffield Laboratory Coking Test. The Effect of Clearance of Plunger on the Swelling Power of Coal in a Steel Retort 345
CXXXII. Sheffield Laboratory Coking Test. The Effect of Dropping a Steel Plunger Through Different Heights on the Density of the Coal Charge . 347
CXXXIII. Sheffield Laboratory Coking Test. Swelling Phenomena of Compressed Coal 347
CXXXIV. Sheffield Laboratory Coking Test. Comparison of the Swelling Power of Different Coals in Silica and Steel Retorts 348

TABLE NO.		PAGE
CXXXV.	Analysis and Swelling Power of Coals . *facing p.*	352
CXXXVI.	Exceptions to the Limits Imposed in Fig. 118 for Coals in Table CXXXV	354
CXXXVII.	Comparison of Coarse and Fine Crushing . .	374
CXXXVIII.	Analyses of Coals of Table CXXXVII . . .	377
CXXXIX.	Comparison of Top-charging and Cake-charging a Derbyshire Coal	385
CXL.	Cokes made from Derbyshire Coals . . .	385
CXLI.	Comparison of Cake-charging and Top-charging Yorkshire Coals	387
CXLII.	Comparison of Methods of Charging for a Yorkshire Coal (No. 23 C and D)	388
CXLIII.	Varying Rates of Coking for Ovens of Different Widths	393
CXLIV.	Effect of Variation of Coking Time for Parkgate and Silkstone T.P. Slack	398
CXLV.	Effect of Variation of Coking Time for Parkgate Coal	399
CXLVI.	Effect of Variation of Coking Time for Barnsley B.M. Coal	400
CXLVII.	Effect of Variation of Coking Time for Derbyshire Coal	400
CXLVIII.	Effect of Variation of Coking Time for West Yorkshire Mixture G.	401
CXLIX.	The Effect of the Rate of Heating on South Yorkshire Mixture R.	402
CL.	Effect of Variation of Coking Time on Parkgate Coal	404
CLI.	Blending Parkgate and Silkstone Coals . .	407
CLII.	Blending Parkgate and Halifax Soft Coals . .	409
CLIII.	Blending West Yorkshire Mixture with Parkgate Coal	411
CLIV.	Analyses of Constituent Coals of Derbyshire Mixture No. 45	413
CLV.	Blending Derbyshire Coal with Better Coking Coals	414
CLVI.	Size Analyses of Coals Used for Tests in Table CLV	415
CLVII.	Blending Barnsley B.M. Coal with Parkgate and Silkstone Coals	418
CLVIII.	Blending Barnsley K. with Parkgate R.M. . .	421
CLIX.	Blending Barnsley R. with Parkgate-Barnsley D. .	423
CLX.	Blending Barnsley H.M. with Other Coals . .	425
CLXI.	Effect of Adding Durain and Fusain to Parkgate Bright Coal	427
CLXII.	Effect of Blending Durain with Parkgate M. Coal	428
CLXIII.	Effect of Vitrain on the Quality of Coke . .	429
CLXIV.	The Effect of Blending Durain with Barnsley H.M. Bright Coal	430
CLXV.	The Effect of Durain when Coking Nut Coal .	432
CLXVI.	The Coking of Parkgate and Silkstone Beans of Varying Durain Content	433
CLXVII.	Blending Fusain with Parkgate and Silkstone Beans	435

TABLE NO.		PAGE
CLXVIII.	Effect of Adding Fusain to Parkgate and Silkstone Slack	436
CLXIX.	Effect on Shatter Indices of Cokes of Adding Varying Amounts of Fusain to Different Slacks	437
CLXX.	The Addition of Fusain to "Non-Coking" Coals	438
CLXXI.	Effect of Adding Fusain to a Blend of Non-Coking and Coking Coals	438
CLXXII.	The Effect of Blending 5 per cent. of High-Temperature Coke of Different Graded Sizes with Parkgate M. Coal	439
CLXXIII.	The Effect of Blending 5 per cent. of High-Temperature Coke of Different "Through" sizes with Parkgate M. Coal	439
CLXXIV.	The Use of 20-mesh High-Temperature Coke Dust for Blending	440
CLXXV.	The Effect of High-Temperature Coke Dust Through 200-mesh Compared with that of Fusain	441
CLXXVI.	Comparison of the Use of Coke Dust of 20-mesh and 200-mesh Size	441
CLXXVII.	The Effect of Coke Dust of 20-mesh and 200-mesh Size and Fusain	442
CLXXVIII.	Comparison of the Use of Different Coke Dusts with Derbyshire Coals	443
CLXXIX.	Effect of Adding 3 per cent. of Coke Dust to Barnsley Coals	444
CLXXX.	Effect of Adding Coke Dust to Parkgate and Silkstone Slack	446
CLXXXI.	An Attempt to Find a Substitute for Fusain	447
CLXXXII.	The Effect of the Fineness of Coke Dust on the Abradability of Coke	447
CLXXXIII.	Effect of Fineness of Coke Dust on the Abradability of Coke	448
CLXXXIV.	Increase in 1½-in. Shatter Index on Adding Fusain, Coke Dust and Durain to P. & S. Bright Coal	450
CLXXXV.	Effect of 4 per cent. Addition of Different Dusts on the Impact Hardness and Abradability of Coke	453

PART I
INTRODUCTION

CHAPTER I

STANDARDS FOR COMMERCIAL COKE

"COKE" may be described as the cellular residue from the carbonization of a coking coal in commercial ovens or retorts at temperatures of about 900° C.; and a "coking coal" as a coal which will yield a commercial coke when it is thus carbonized.

Some of the questions arising from these descriptions are: What are the standards required of a commercial coke; what is the cause of coke-formation; what are the characteristics of coking coals and how may they be judged; and how can the quality of coke be improved.

It is proposed to discuss these questions in detail. Before doing so, it is desirable, in this Introduction, to indicate the problems involved, to outline our knowledge of the formation of coke and of the characteristics of coals which influence its quality, and to consider briefly the effects of the conditions of carbonization. For the most part, examples of the effects of the conditions of carbonization will be drawn from by-product coking practice.

The qualities which render a coke most useful, or most readily saleable, vary somewhat according to the use to which it is to be put. For all combustion processes, which account for most of the coke used, it would be expected that the intrinsic "combustibility" of the coke would be important. The manner in which a coke burns depends so much, however, upon such factors as, for example, the size of the pieces and the rate of supply of air to the fuel-bed, that differences in intrinsic combustibility may be masked. It is therefore necessary to consider separately the different combustion processes in which coke is used. From observation of the various factors upon which the usefulness of a coke for particular purposes depends, it should be possible to indicate those qualities of a coke which render it most valuable for all, or most, purposes.

Coke which has been heated to a temperature of about 900° C. during its manufacture has a cellular structure, and contains only a small proportion of "volatile matter." It consists mainly of carbon, with some mineral matter and moisture. The cells are usually from 1 to 3 mm. in size, and are roughly spherical; the cell walls are up to several mm. in thickness. Most of the cells are open. The combustion of coke is smokeless, because it contains hardly any volatile hydrocarbons, and the products of combustion

are carbon dioxide and carbon monoxide. Neither swelling nor caking occurs on combustion, so that coke can be used in a deep fuel-bed without the necessity for frequent poking to cause it to settle, provided that the size of the pieces bears the correct relationship to that of the furnace to avoid "arching." When a bed of coke has been ignited, it should soon attain a red heat and should continue to burn until it is almost all consumed.

The principal combustion processes in which coke is used are: (1) for domestic heating, in open grates; (2) for the convection-heating of buildings, in enclosed slow-combustion stoves; (3) for water-heating and the heating of rooms by direct radiation as well as by convection, in semi-enclosed "domestic" boilers; (4) for the central-heating of houses in small enclosed boilers; (5) for the central-heating of large buildings, in large enclosed boilers; (6) for steam-raising in large enclosed boilers; (7) for minor metallurgical operations, such as forging in braziers or on hearths; (8) for metal-melting in crucibles, in furnaces; (9) for metal-melting, in cupolas; (10) for gas-making, in producers; and (11) for iron-smelting, in blast-furnaces.

Coke for Grates, Stoves and Boilers.—When coke is burnt in open grates, slow combustion stoves, domestic boilers or small central-heating boilers, the rate of combustion per square foot of grate-area is low at certain times. There is then a tendency for the temperature of the fuel-bed to fall so low that active combustion cannot be maintained. Extinction of the fire is particularly liable to occur if the bed of coke does not settle regularly as the lower layers are burnt away; and irregular settlement of the bed may occur if the size of the pieces of coke is too great in relation to the size of the furnace. Accumulation of ash on the grate-bars offers resistance to the passage of air, and, if the draught is unalterable, may reduce the quantity of air that can pass to below that sufficient to maintain the fire; a voluminous ash, or one liable to clinker, offers the most resistance.

In these "domestic" uses of coke, certain of its combustion characteristics are important. Its reactivity, as measured, for example, by its rate of reaction with oxygen under specified conditions, may vary according to the amount burned during the test. There may be present a sufficient proportion of highly reactive pieces (such as are found amongst under-carbonized "black ends") to cause it to ignite readily. Or, *per contra*, many of the pieces may have an unreactive skin of hard carbon (such as may form when gaseous hydrocarbons decompose) which, if burnt away, would expose reactive material beneath. It will be shown that, with respect to the combustion of coke in small stoves, its specific reactivity and its size, together with the temperature of the fuel-bed, must be taken into account. With a coke of given size, the higher its specific reactivity the more readily will it keep alight in the stove for a

long time when burning at a low rate ; and with a given temperature of the fuel-bed, the pieces of coke can be larger the more reactive it is.

Since the temperature of the fuel-bed is liable to fall in all small stoves which it is desired to keep alight without attention (*e.g.* overnight), the size of the pieces of coke and the specific reactivity of the inner portions, when the skin of the cell-walls has burned away, are of most importance. The upper limit of serviceable size is determined by the dimensions of the furnace, and the lower limit by the draught. For a coke within these limits of size, the specific reactivity and the ash-content require to be such that the fire will readily remain alight. The reactivity should not be so high, however, that much reduction of carbon dioxide to monoxide occurs when there is a deep fuel-bed, otherwise there may be waste of fuel or trouble through the escape of the carbon monoxide.

In the normal use of coke in domestic appliances during the day-time, the principal requirement is for the coke to be burnt at a specified rate. The amount and character of the ash as affecting the radiant efficiency of the fuel is important only with open grates or partially closed stoves. With closed stoves, the ash-content of the coke affects its performance if clinker is liable to be formed. In general, it may be said that most attention should be paid to the size of the coke used in small closed stoves and in open grates, whilst in the latter the specific reactivity of the coke is also of considerable importance.

In large boilers for central heating, and in coke-fired ovens and furnaces, the grate-areas may be considerably larger than is necessary in domestic stoves, and such characteristics of coke as influence its regular combustion over the whole of the grate-area assume importance. Ease of ignition does not matter much, though the size of the coke should be such that the production of a bright fire is not unduly delayed. Actually, the design of the grate-bars probably matters more than the character of the coke as regards the maintenance of a uniform fire.

Coke for Hearths and Braziers.—In the use of coke on a hearth, as in forging, its size must be chosen in relation to that of the article to be heated and must admit of the charge being heaped around and over the article, so as to cover it. During the forging of small articles, such as knife-blades, the operator has to stand close to the hearth. A coke which " spits " or flies into small pieces is therefore objectionable. Pieces of shale embedded in the coke are most liable to fly when heated, so that the coke chosen for use on a hearth should be made from a well-cleaned and finely-crushed slack. The coke fire should maintain its heat reasonably well after the blast has been turned off from the hearth, a requirement which causes the specific reactivity of the coke to be taken into account.

Coke for Metal Melting.—In melting operations, the requirements differ according to the temperature to be attained. For tin, lead, zinc and aluminium, the melting-temperatures range between 250° and 650° C; for brass, silver and copper between 850° and 1100° C.; and for steel, up to 1600° C. or more. No difficulty is experienced in attaining the temperatures necessary if the size of the coke is suitably chosen. For crucible-steel melting, the high temperatures required, and the necessity for maintaining that temperature until the fuel bed has burnt down to a low level, demand special qualities in the coke which appear to be best met by "beehive" cokes, which are amongst the least reactive. A high enough temperature can only be obtained economically with an unreactive coke which does not clinker.

Coke for Cupolas.—In foundry iron cupolas, the fuel-bed has a greater depth as compared with the fuel-beds hitherto considered. In small cupolas, a large coke cannot descend regularly and it is desirable to use coke of graded size. In large cupolas the size of the coke is not of so much moment, but large coke avoids loss by "solution" in the shaft. The prime consideration is the production of a high temperature with economy in coke-consumption. To this end, a hard unreactive coke which does not produce breeze on handling or during its passage through the cupola should be used. Purity, particularly as regards sulphur-content (which should be less than 0·8 per cent.) and ash-content (to reduce the volume of slag), is essential.

Coke for Gas-Producers.—For use in gas-producers, as, for example, at gas-works for heating the retorts, the coke need have no special qualities, though the ash it contains should preferably have a high melting-point. Cokes in general have the principal quality that a producer-fuel should possess, namely, the ability to pass down the shaft of the producer without swelling or caking, and they can therefore be gasified at a high rate per square foot of grate-area unless they contain much breeze. They should not be so soft as to form breeze within the producer. In water-gas generators, two distinct operations are alternated, namely the "blow," during which a blast of air is used to raise the temperature of the fuel-bed, and the "run," during which steam is passed until the temperature falls as low as is consistent with the economic carrying out of the "water-gas reaction." It is desirable that the "blow" should be as short and the "run" as long as possible. A highly reactive coke should therefore be advantageous, so that the fuel-bed shall quickly attain a high temperature during the blow, and so that the run can be carried to a low temperature. The ash of the coke should have a high melting-point.

Coke for Blast Furnaces.—In blast-furnaces, the bed of coke (mixed with the other materials of the charge) is deeper than

in all other operations in which the combustion of coke is effected. The attainment of a high temperature in a comparatively small zone of the furnace is the first essential. This is dependent on the rate of combustion of the coke per square foot of hearth-area, and on the size and specific reactivity of the coke, the size of the combustion zone being governed mainly by the size of the coke. It has been argued that a coke of high specific reactivity is desirable to maintain a small oxidation-zone, but, at the high temperatures obtaining, the small differences in reactivity observable amongst commercial cokes are unimportant. At the high temperature developed in the hearth, the rate of combustion of the coke is not limited by the rate of reaction between oxygen and carbon but is determined by the rate of supply of air. The rate of supply of air is affected by the resistance of the burden in the furnace, and this in turn is affected by the nature of the slag (a basic slag being pasty), and by the manner in which the burden packs, as determined, for example, by the amount of small coke and small ore present. It has been found that the blast-pressure necessary to pass a given amount of air into a furnace can be reduced by screening out breeze from the coke. Such a method of improving the performance of a blast-furnace, however, only takes account of the breakage caused during the handling of the coke up to the point of its discharge into the furnace. More breeze is formed during charging and during the descent of the charge. It would seem reasonable, therefore, to guard against the formation of breeze by using a hard coke. Hardness, in fact, would seem to be the dominant physical property that a blast-furnace coke should possess.

The "hardness" of a coke comprises its resistance to shattering and its resistance to abrasion. The resistance of a coke to shattering can be measured by the so-called "shatter test," in the course of which some indication is obtained also of its resistance to abrasion, which may also be measured by a separate test. Commercial blast-furnace cokes are not readily abradable, unless they have been made from mixtures of coals which include abnormal proportions of non-coking coals.

The Most Useful Qualities of a Coke

From the foregoing brief discussion of the various uses to which coke is put, it will be realised that, whilst there is some variation in the requirements according to the particular use contemplated, certain qualities in a coke stand out as being always desirable. Purity, for example, is always an asset, and often the character of any impurity (*e.g.* the bulk density or the fusibility of the ash) has to be considered. The size of the pieces of coke almost invariably must be chosen in relation to the character of the furnace in which the coke is to be used. The quality of hardness is essential in cokes to be used in deep fuel beds.

The quality of "combustibility" of a coke is not easy to define

or assess, because the behaviour of a coke on combustion is modified by the conditions of its use and is markedly affected by its size. Similarly, measurements of the specific "reactivity" of cokes may not give a true comparison of their usefulness. Laboratory tests have been standardised for measuring, for example, the amount of decomposition of carbon dioxide by coke at 900-1000° C., or the rate of reaction of coke powder with oxygen at about 300° C., or the rate of supply of air required to keep a small charge of coke alight. In all such tests the temperature of the coke is lower than is normal for industrial operations and, although it may be possible by these means to correlate the behaviour of a finely-divided coke with that of the same coke in larger pieces as regards its ability to light quickly and to remain alight, its behaviour under conditions of slow combustion does not necessarily bear a constant relation to its performance when incandescent.

Although a characteristic quality of coke is its cellular structure, measurements of the degree of porosity have not suggested that they can provide standards for commercial cokes which, indeed, do not differ much in this respect one from another. The percentage volume occupied by pores can be measured, and the proportions of "open" and "closed" pores can be distinguished.

Following these considerations, we can now outline the qualities of coke which most demand assessment.

Purity.—For all uses of coke, the quantity of impurities that dilutes its main constituent, carbon, should be as little as possible. The principal impurities are, water, ash and sulphur, whilst the small quantities of phosphorus and arsenic present may render a coke unsuitable for certain purposes.

The assessment of all these impurities requires considerable care, more particularly when obtaining a representative sample of a consignment to be tested, and when reducing that sample to a size convenient for analysis. Later in this book, suitable methods of sampling and analysis of coke will be described.

Size.—In general, a fuel-bed settles more regularly, and combustion is more uniform, the closer the sizing of the pieces of coke, and the most satisfactory range of size, as well as the most satisfactory size itself, is usually smaller the smaller the scale of operation. For example, a satisfactory range of size for a domestic boiler may be ½-in., the size of the pieces being, say, ¾ to 1¼-in., whereas for larger boilers a range of 1-in. may suffice, the size of the pieces being say, 1¼ to 2¼-in. The actual screening range and size chosen will depend to a certain extent on the "combustibility" of the coke, and on the way in which it breaks when the sizes are being prepared by "cutting" large coke. For example, a fingery coke tends to yield a high proportion of prismatic pieces with their length twice or thrice as great as their cross-section; such a coke should be graded over as small a range as is practicable.

In the operation of grading coke, it is usually screened over round holes, whereas tests of the graded material are made over square-mesh screens. Assuming the coke pieces to be approximately cubical, as is most often the case, the theoretical relationship between the two types of screens is $d = \sqrt{2}l = 1{\cdot}414l$, d and l being the diameter of the round hole and the side of the square hole respectively. Actual tests, with coal, of round hole and square mesh screens have shown the ratio of the size passed by the two types to be about 1·15 to 1.

Different types of screens for the same nominal sizes of coke may give different grades, either because the apertures are of different shapes or because of differences in the motion imparted to the coke during screening. A reliable standard of comparison of the size of cokes can only be made by hand-screening with meshes of standard size and pattern. In subsequent references to the size of coke, unless otherwise stated, the square mesh size is used.

Hardness.—Oven coke is sometimes referred to as "hard" coke, to distinguish it from the softer coke that is made, for example, in gas-retorts. The quality of hardness then considered is that of the cell-walls, *i.e.* the "surface hardness" or abradability of the coke. The surface hardness of gas coke is often low if it has been made from coarse slack, say through 2-in., in horizontal retorts, or from nut coal, say 1 to 2-in., in vertical retorts. The greater surface hardness of an oven coke is due to its having been made from crushed coal, through $\frac{1}{4}$-in. or even $\frac{1}{8}$-in., in tall ovens, providing a greater bulk-density and a greater uniformity of the charge than in a gas-retort; the voids between the particles of coal being small, there is but little opportunity for local swelling of individual particles to produce zones of spongy coke of low surface hardness.

Surface hardness being an essential quality of an oven coke with respect to its principal use (for blast-furnace and foundry practice), cokes of low surface hardness are not often purposely made in ovens, or, if made, are not regarded as commercial metallurgical cokes. It is only from coking coals of the lowest rank, or from mixtures containing an excessive proportion of a poorly-coking or non-coking constituent, that cokes of low surface hardness are produced in ovens.

The "impact hardness" of a coke, on the other hand, is usually its most valuable physical property with respect to its use in blast-furnaces and cupolas; and it is convenient to use the simple term "hardness" of an oven coke to denote its impact hardness or resistance to breakage by impact. The hardness of coke can be measured by the "shatter test" (*British Standard Specification for the Sampling and Analysis of Coke, No.* 496), in which 50 lb. of coke over 2-in. in size are dropped four times from a height of six feet on to a metal plate and a screening analysis is then made of the shattered material.

Combustibility and Specific Reactivity.—If the "combustibility" of a coke be defined as its ease of combustion under specified conditions, it will be understood that measures of that property may be misleading. For the ease with which a coke burns is markedly influenced by the conditions under which it is burnt, and the particular conditions of use contemplated may not be comparable with the "specified conditions" of test. Some of the factors which influence the burning of a coke in a small stove, for example, are the size of the coke, the size of the stove, the bulk-density of the fuel-bed, the draught, the volume and degree of fusibility of the ash and the specific reactivity of the coke. If cokes of a closely-graded size and of normal ash-content are used, their behaviour in a given stove will be determined mainly by their specific reactivity.

The specific reactivity of a coke can be measured by its rate of reaction with oxygen under standard conditions.

CHAPTER II

STAGES IN THE FORMATION OF COKE

A "COKING" coal can be differentiated from a "non-coking" coal by the fact that, when it is heated, it softens, usually within the range 350° to 400° C. and then re-solidifies, usually at about 500° C., with a general cellular structure. Coals which are non-coking in the commercial sense may yield residues on heat-treatment which, though partly cellular, are insufficiently so for the product to be of commercial value. In this respect much depends on the size of the coal. For example, the small coal (slack) of the Thick seam of South Staffordshire is "non-coking," but a commercial coke, a "hearth" coke, was formerly extensively made from large lumps of this coal by partially burning heaps of it, covered by a layer of coke dust, in the open air. Such a hearth coke is of a low degree of hardness, fragile and abradable.

In general, the distinction between a coking and a non-coking coal is of moment commercially only as regards the small coal commonly used for the manufacture of coke in ovens and retorts. It will be understood, therefore, that the criterion of a coking coal, that is, that its carbonised residue must have a cellular structure, applies not only to each individual particle but to an agglomeration of such particles. For the commercial product must be massive. The unagglomerated residue obtained on heating a small coal that is non-coking is sometimes termed a "char." Agglomeration of the particles of coal when a commercial coke is formed is usually so complete that their shape is lost. Sometimes, however, the pieces of coke may have a rough surface due to the presence there of unagglomerated or partially-agglomerated pieces. Such a coke is abradable and is rarely acceptable as a "furnace" coke.

In a charge of small coal in an oven, in practice, there always remains not less than about 25 per cent. of free space, no matter how close a packing may be given to the particles by choice of size, by pressure as in a stamped charge, or by falling through a height, as in top-charging. Unless the individual particles are capable of swelling by at least 25 per cent. by volume, the charge will not become thoroughly agglomerated when it is coked. Ability to swell is therefore a primary requisite of a coking coal.

The swelling of a piece of coal is effected by the evolution of volatile matter from it whilst it is plastic. The plasticity of coal

is a complex phenomenon which appears to be induced primarily by the pressure of gases causing surface flow at the moment when, under the action of heat, the molecules at the surface have attained a degree of freedom comparable with that obtaining in a liquid. Later in this book, evidence will be given to show that the plasticity of coal does not precede the evolution of "volatile matter."

Whilst the plasticity and swelling of coal and the formation of coke are intimately connected with the evolution of volatile matter, the evolution of volatile matter *per se* cannot determine the formation of coke. There must be sufficient resistance offered to the escape of the volatile matter in the gaseous state to develop a pressure high enough to cause surface flow and to form a porous structure. Such a resistance is afforded mainly by films of oil and tar (part of the "volatile matter") adsorbed at cracks and partly-formed pores so as to seal them. The adsorption of oil by the coal is thus an essential if a coke is to be formed. The ability of a coal to adsorb oil depends upon its rank. A coal of low rank (and high oxygen-content) adsorbs oil less readily than one of high rank (and low oxygen-content). With a coal of low rank the oils produced on heating for the most part distil away without being adsorbed. Partly-formed pores and cracks then remained unsealed and the volatile matter escapes freely without developing a uniform porous structure in the residue.

During the heating of a charge of a coking coal, the successive stages are (1) pre-softening, (2) decomposition and softening, (3) swelling, (4) agglomeration, (5) solidification and (6) hardening.

Pre-Softening.—Some decomposition of the reactive groupings of the ulmins in the coal conglomerate occurs during the pre-softening stage, yielding small quantities of carbon dioxide and steam, whilst resins and hydrocarbons disseminated throughout the conglomerate are liberated. Solvent analysis of a coking coal that has been heated to about 325° C. (*i.e.* below the temperature of softening) shows that from 2 to 4 per cent. of an oily extract can be obtained with ethyl ether, whereas the unheated coal yields but a trace of extract to that solvent. This oily extract comprises resins and hydrocarbons liberated from the coal conglomerate but absorbed within it.

Decomposition and Softening.—Active decomposition of the coal as a whole begins during the softening stage, and gaseous and liquid hydrocarbons are released. The liquid hydrocarbons are readsorbed, partly on the surfaces of the particles from which they have been evolved, but mainly on the surfaces of adjacent particles. The mass of coal then becomes sufficiently plastic to be distorted, due to the adherence and partial agglomeration of individual particles, if external pressure be applied, but the degree of distortion is small unless the temperature approaches that at which swelling begins. This softening of the mass of particles has been found to

accompany incipient swelling of the individual particles which, if examined separately, are found to possess a minute cellular structure.

Swelling and Agglomeration.—Whilst swelling of individual particles begins during the "softening" stage, the free space between them has to be completely occupied or enclosed before swelling of the mass of coal as a whole is observed. When this general swelling begins—it is the stage most readily determined by laboratory test—large closed pores are formed, mainly by the enclosure of the "free spaces."

The degree of swelling of the mass of coal naturally is governed by external conditions. In the "crucible" test for volatile matter, in which a small quantity of coal is used, the external pressure is negligible and a swollen and porous "button" can be produced. Under small load, the charge of coal may swell to four or five times its original size, but a sufficiently heavy load, say 700 lb. per sq. in., can suppress this swelling altogether. The forcible prevention of swelling (which is caused, in part, by the evolution of gases) hinders the evolution of volatile matter, which may be delayed until, at a higher temperature, the plastic mass solidifies. The gases then escape through the rigid cellular structure of inter-connected pores.

In coke-making practice, the extent to which the ability of a coal to swell can be exercised is of considerable importance. When the charge in a coke oven is heated from both sides, two vertical "plastic layers" are formed which move gradually towards the middle. In the main mass of the charge, the tendency of any one coal particle to swell is neutralised by a similar tendency of particles above and below it, but at the top of the oven the swelling is not thus restricted and the coke formed there may be spongy. The swelling of the charge in contact with the walls of the oven is often made manifest by the imprint of the mortar spacings of the bricks on the resultant coke. With most coals, however, there is a marked shrinkage of the charge as the rigid coke is heated to the highest temperature of carbonisation, and it becomes detached from the walls so soon as it ceases to be plastic. Should the coke not shrink sufficiently, as may be so with the cokes from some low-volatile coals, dangerously high pressures may obtain and the walls of the ovens may suffer damage. In general, the most strongly swelling coals, such as the Durham coals, make the hardest cokes, partly, no doubt, because the pressure developed during carbonisation effects uniform agglomeration of the individual particles of coal.

In a horizontal gas-retort which is not fully packed, a plastic layer of elliptical contour is formed and a coal that is too strongly swelling may form too spongy a coke. For this reason, coals of moderate swelling power are most commonly used. In continuous vertical retorts, the plastic layer is also of elliptical shape, and a strongly swelling coal may offer too high a resistance to the continuous flow of coal through the retorts.

Solidification and Hardening.—At about 500° C., or at a rather lower temperature if there is freedom to swell, the plastic coal begins to solidify and a rigid cellular structure is formed. Gases evolved subsequent to this solidification escape through the network of open pores. There is a considerable loss of volatile matter (mostly gaseous) after solidification, and this loss causes the structure to shrink. Cracks are formed which, if of large size or considerable number, weaken the coke. Control of the hardness of coke is effected mainly by controlling the degree or character of this shrinkage.

CHAPTER III

THE CHARACTERISTICS OF COALS WHICH INFLUENCE THE FORMATION OF COKE FROM THEM

FROM a consideration of the stages in the formation of coke it will have become apparent that the factors most concerned in the production of a hard coke from a coal are the ability of the coal to swell when heated, the ease with which the oils distilled from it can wet the surfaces of the particles in the charge, and the extent to which loss of volatile matter produces shrinkage cracks in the solidified structure.

Another factor, only hinted at hitherto, requires consideration, namely, the production of sufficient oil during the early stages of heating to seal the incipient pores and to resist the escape of gases, to such an extent that swelling of the particles can occur. Lack of sufficient oil distilled from "low-volatile" steam coals and anthracites renders them non-coking, whilst the non-coking coals of high volatile matter content yield oils which are incapable of wetting the particles sufficiently.

THE ULTIMATE ANALYSIS OF COALS

All the characteristics of coals upon which their coking-power depends can conveniently be expressed in terms of their ultimate analysis, for there are relationships, direct enough for practical purposes, between the carbon and hydrogen contents of the coal substance and its swelling-power, its "wettability" to oils, its volatile matter content and its oil-producing capacity. These relationships will be discussed later.

The hardest cokes are yielded by coals of high carbon-content (89 to 91 per cent. dry, mineral-matter free basis) and medium hydrogen content (4·6 to 5 per cent.); such coals are of low oxygen and volatile matter contents. Their swelling-power and "wettability" are high, and the comparatively small quantity of volatile matter evolved from them after plasticity has ceased ensures that the cokes they yield shall not be unduly weakened by shrinkage cracks.

A coking coal of 91 to 89 per cent. carbon-content may conveniently be termed a coking coal of "high rank," whilst coking

coals of 89 to 87, 87 to 85 and 85 to 83 per cent. carbon-content may be termed of "moderately high," "medium" and "low" rank respectively.

The most reactive cokes are formed from coals of low carbon-content, partly because such coals are initially highly reactive and this property is retained in the carbonised residues, and partly because the particles in the charge are not effectively wetted by the oils and tars distilled during carbonisation and a protective coating of unreactive tar-carbon is not formed on the surface of the coke.

A property of coal which may adversely affect its value for coke-making is its ability to combine with oxygen during "weathering." With many coals, notably those of high swelling-power, there is insufficient oxidation during storage in the open air, during a year even, to alter their properties. With coals of low rank, however, their swelling-power and their ability to yield a strong coke may be seriously impaired by exposure to air for a longer time than normally elapses between their being mined and charged in the ovens. It is unwise to store such coals for long, particularly if their size is small.

The Blending of Coals

Blends of two or more coals of different ranks in different proportions do not yield cokes with properties equivalent to the proportion of each coal in the blend. It is possible to use 50 per cent. of a coal which is, by itself, non-coking if blended with a suitable coal. On the other hand, the addition of but 20 per cent. of one coal to another may provide a coke of lower impact hardness than is given by either of the coals singly. It is difficult to generalise on the results of blending coals for coke-making, and this subject is discussed in detail in a later chapter.

CHAPTER IV

THE INFLUENCE OF CARBONISING CONDITIONS ON THE FORMATION OF COKE

THE quality of the coke produced from a given coal can be affected to a considerable extent by the conditions of carbonisation.

The manufacture of coke originated in Great Britain with the partial burning of large lumps of feebly-swelling or non-swelling coals in piles in the open air and developed later, where conditions permitted, into the carbonisation of small coal of medium or moderately high rank in "beehive" ovens. These methods of coke-manufacture were copied on the Continent, together with British methods of iron-smelting, but did not prove entirely suitable for the high rank coals available, which were found to be more successfully treated in narrow retort-ovens. Later, the by-product oven was developed from the retort-oven and introduced in this country.

During the past thirty years there have been considerable changes in the general design of by-product ovens, which initially did not produce as good cokes as were provided by the beehive ovens they displaced. As compared with the earlier ovens of dimensions 33 ft. long, 7 ft. high and 21 in. wide, built of semi-silica brick and charged usually with compressed coal, modern ovens are usually of silica brick and may be 40 ft. long, 13 ft. high and 18 in. wide, taking a loose charge of coal. The heating of the ovens has been made more uniform and, in consequence, a more uniform product than was possible from the older types of ovens is obtained.

The principal conditions of carbonisation affecting the quality of the coke produced are (1) the size of the coal, (2) the bulk-density of the charge and (3) the rate of heating, in which connexion the width of the oven must be considered.

The Size of the Coal.—Except for coals of the highest swelling-power, with which such high pressures are developed that large pieces in the charge lose their identity, coarse particles tend to yield a coke of irregular structure. Coke of the most uniform structure is produced from a finely-divided charge, such as that given by fine coal cleaned by froth-flotation, but the finer the grinding of the coal the lower, in general, is the bulk density of the charge, a matter of considerable importance when the oven is top-charged.

The Bulk Density of the Charge.—The bulk-density of the charge is affected by the proportions of different sizes of coal present, by the moisture-content and by the height through which the charge is dropped; or, when a stamped charge is used, by the pressure applied to the cake. The quantity of coal that can be introduced into an oven depends on the bulk-density of the charge, which thus affects the rate of carbonisation.

If a coal has been top-charged, it yields a coke with fewer fractures than when it is charged as a compressed cake because, its bulk-density being less, the amount of volatile matter awaiting release, after plasticity of the charge has ceased, is less per cubic foot, and the amount of shrinkage is less. A coke made from a compressed cake may look stronger than one from the same coal that has been top-charged, and its surface hardness may be higher, but the greater number of shrinkage cracks throughout its mass causes it to be liable to break into small pieces when it is dropped.

The Rate of Heating.—For a given flue temperature, and with a given charge of coal, the coking-time is directly proportional to the square of the width of the oven. Thus, assuming that for a 21-in. oven, with a flue-temperature of 1100° C., the coking time is 36 hours, the following coking-times should apply to other widths of ovens:

WIDTH OF OVEN	Inches	20	18	16	14
COKING-TIME	Hours	32½	26½	21	16

The use of silica has enabled considerably higher temperatures than 1100° C. to be used, and the coking-times to be correspondingly decreased. Thus with a flue-temperature of 1400° C., the coking-times for different widths of ovens would be:

WIDTH OF OVEN	Inches	21	20	18	16	14
COKING-TIME	Hours	24	22	17½	14	11

The rate of heating is naturally related to the coking-time. The most important period is during the plasticity of the coal, over the temperature-range 400° to 500° C. Taking the wider range of temperature 300° to 600° C. so as to include this important period, the average rate of rise of temperature in a 20-in. oven coking in, say, 30 hours is 1·0° C. per minute at ¼-width, whilst in a 14-in. oven coking in 11 hours the average rate of rise of temperature at ¼-width will be 2·5° C. Such variations in the rate of heating are not great, in comparison with the rates of heating obtainable in laboratory tests, but they have important effects.

Differences in the rates of heating at different zones across the oven are perhaps as important as differences in the average rate of rise of temperature. When the charge is coked slowly, the rate of heating at the walls of the oven and at the centre may differ

considerably, and the coke will in consequence lack uniformity. No doubt this is why a coal of low rank yields a coke of irregular shape in wide ovens, whilst its quality can be improved by increasing the rate of heating, a matter fully discussed in our first report ("Coke for Blast Furnaces," London, 1930). A modern narrow oven is more successful than the older ovens in producing a hard coke from a coal of low rank.

Certain coals appear to be remarkably sensitive, with respect to their ability to yield a good coke, to changes in the average rate of heating, so that a rate neither too fast nor too slow must be used for them. Cokes made in 14-in. ovens from strongly-swelling coals of high volatile matter content are, in general, not so strong as cokes made from the same coals in 18-in. ovens under modern conditions, but it is possible, by suitable blending, to overcome the tendency that some coals possess to yield a spongy coke at the centre of the oven.

PART II

THE STANDARDS OF A COMMERCIAL COKE

CHAPTER V

THE SIZE OF COKE

THE size of coke was considered in our first report (Part I, Chapter II), and the opinion was advanced that, whereas coke of size greater than 3 in. is of no special advantage in the blast furnace, it is of considerable importance that too small a size should not be charged, and that $1\frac{1}{2}$ or 1 in. is probably the minimum useful size. It was shown that the size of coke as discharged from the ovens is influenced by the quality of the coals and their size, by blending of the charge, by the method of charging the ovens and by the rate of heating. During the handling from the ovens to the furnace the amount of dropping that the coke receives affects its size (to a degree dependent on its hardness), but breakage by attrition or abrasion within the furnace should be negligible.

Further information on this subject can now be given.

In blast-furnace trials carried out for the Midland Coke Research Committee, the coke in the skips for charging a furnace contained only about 3 per cent. over 4 in. and about 16 to 21 per cent. over 3 in. in size (*J. Iron and Steel Inst.*, 1933, **1**, 87). From tests designed to simulate the dropping of the coke from the skips to the charge level of the furnace, it was deduced that there then remained only about 1 per cent. over 4 in. and about 6 to 8 per cent. over 3 in. Nevertheless, for a burden, other than coke, of 74 cwt. per ton of pig, the consumption of coke was only 25 cwt. and the slag-formation 23 to 24 cwt.; a notable performance when making No. 3 foundry iron from lean ores.

In the same series of trials, the coke sent to the furnace was screened before loading it to the charging skips, so as to remove, in successive trials, breeze of under $\frac{3}{4}$-in., $1\frac{1}{4}$-in. and $1\frac{1}{2}$-in. sizes. There was a reduction in the coke consumption of 2 cwt. per ton of pig when $1\frac{1}{4}$-in. breeze was removed. Whilst no further advantage was obtained by removing $1\frac{1}{2}$-in. breeze, it may be desirable to remove the larger size in practice because it is more readily saleable after re-screening. The generally beneficial effects of removing the breeze were pronounced. The blast-pressure required to pass a given quantity of air was much reduced, a higher blast-temperature could be used and there was a reduced top-gas temperature (to as low as 150°F.) and a reduced ratio CO/CO_2 in the top gases. The furnace could be driven faster and was less subject to derangement,

These effects of removing breeze are illustrated in Fig. 1 whilst in Table I are recorded the results obtained when, over several periods of working the same furnace, different quantities of $1\frac{1}{2}$-in. breeze were screened from the coke.

TABLE I.—THE EFFECT ON COKE CONSUMPTION IN A BLAST-FURNACE OF REMOVING DIFFERENT QUANTITIES OF BREEZE.

Period	Breeze removed, per cent.	Pig made. Tons per week	Burden. Cwt. per ton of pig	Slag. Cwt. per ton of pig	Dry Coke used. Cwt. per ton of pig	Dry Coke used. Corrected.* Cwt. per ton of pig
July 28, 1930–Dec. 7, 1930	2·0	707	75·8	22·9	26·3	26·3
Jan. 4, 1931–July 26, 1931	3·2	672	73·4	23·7	26·6	26·3
July 26, 1931–Aug. 23, 1931	5·6	664	72·5	24·2	27·1	26·7
Aug. 23, 1931–Sept. 20, 1931	8·8	761	74·5	24·0	24·7	24·3
Sept. 30, 1931–Oct. 11, 1931	10·6	782	74·2	23·1	25·0	24·9
Oct. 11, 1931–Oct. 9, 1932	13·0	775	68·9	23·0	24·4	24·4

* A uniform slag-production of 22·9 cwt. per ton of pig is assumed, and an allowance is made of 0·33 cwt. of coke per cwt. of slag.

These results indicate that no advantage was gained by screening out the coke breeze until between 6 and 9 per cent. had been thus removed.

The fact should not be overlooked that these records show the result of screening coke immediately before it is charged to the furnace skips. Screening the coke at the ovens may not be equally effective, because the subsequent handling may be such as to produce much breeze.

THE SIZE OF COKE FROM THE OVENS

The size of coke discharged from an oven is no longer a reliable guide as to its hardness, for in modern ovens, with rapid coking, hard cokes of small size may be produced. When, however, different coals are carbonised under standard conditions of heating, the better coking coals, *i.e.* those producing the hardest cokes, usually yield the largest pieces.

The Effect of the Quality of the Coal.—The effect of the quality of the coal on the size of the coke is illustrated by the result of tests made in the experimental oven of the Midland Coke Research Committee (*see* "Coke for Blast Furnaces," p. 232). For a number of different coals, all carbonised under the same conditions, the average size of the whole charge of coke removed

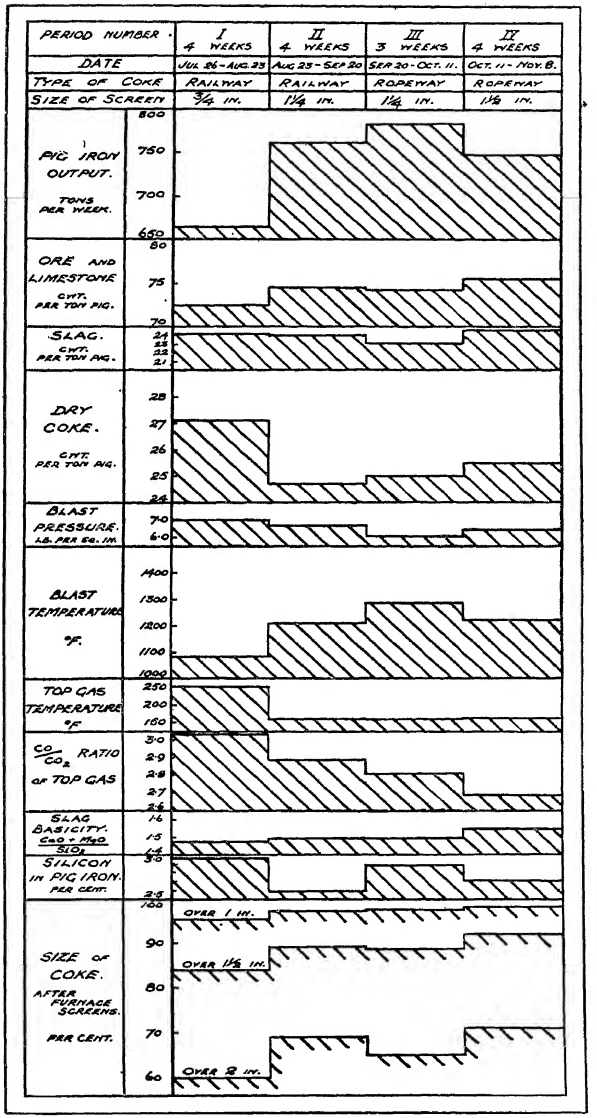


FIG. 1.—The Effect of Screening out Breeze of Different Maximum Size on the Operation of a Blast Furnace. Average Monthly Data.

from the oven is recorded in Table II. Each coal charged was through ¼-in., and the coking time was 18 to 19 hours.

TABLE II.—THE EFFECT OF THE QUALITY OF THE COAL ON THE SIZE OF COKE

(Tests in an Experimental Oven)

Coal	Ultimate Analysis (dry, ash-free basis)		Swelling Power.* %	Size of Coke, per cent. on:				Shatter Indices	
	Carbon %	Hydrogen %		4-in.				1½ in.	½ in.
Halifax Soft	85·9	5·42	109	70	86	96	98	89	97·1
Silkstone (C)	85·3	5·69	179	62	86	92	96	82	95·8
Parkgate (MM)	84·8	5·30	93	72	87	95	97	83	96·1
Parkgate (C)	84·6	5·20	123	74	87	96	97	85	96·7
Parkgate (R)	84·4	5·30	186	66	86	94	97	86	96·7
Barnsley (M)	83·6	5·42	75	58	77	93	96	82	96·9
Barnsley (K)	82·7	5·21	nil	44	64	84	88	84	93·2
Barnsley (B)	82·7	5·55	nil	33	61	86	91	75	94·4
Barnsley (R)	82·0	5·12	nil	35	55	78	85	73	91·9

* Sheffield Laboratory Coking Test.

All the coals yielding cokes that were not readily abradable (*i.e.* with ½-in. shatter indices greater than about 96) produced 96 per cent. or more of coke greater in size than ½-in. With the abradable cokes (*e.g.* that from Barnsley K coal), there was a marked increase in the amount of ½-in. size, whilst the amount of 3-in. and 4-in. size decreased to a disproportionate degree, so that the average size of the coke was small.

All the coals that produced good cokes were of carbon-content greater than 83 per cent., and had marked swelling-power as measured by the Sheffield Laboratory Coking Test.

The Effect of the Size of the Coal.—The results of tests showing the effect, on the size of the coke produced, of grading the size of the coal charged to the ovens, were published in our first report (*see* p. 22). They indicated a decrease in the size of the coke with increase in the size of the coal, the larger particles of coal apparently causing the structure of the coke to be irregular. The effect of the graded size of the coal was most marked when it exceeded ¼-in. In coking practice it is more important to assess the effect of using "through," as distinct from "graded," fine, medium and coarse coals.

Tests made in the experimental oven show that fine grinding, in general, tends to reduce the average size of the coke, though the amount of breeze through ½-in. is reduced. Typical results are recorded in Table III.

TABLE III.—THE EFFECT OF FINE GRINDING OF COAL ON THE SIZE OF COKE.

Coal		Size of Coke, per cent on :				Shatter Indices	
		4-in.	3-in.	1½-in.	½-in.	1½-in.	½-in.
Parkgate and Silkstone T.P. Doubles	Coarse	72	85	95	98·2	78·5	95·5
	Fine	46	75	95	97·9	82·5	97·3
Parkgate and Silkstone T.P. Slack	Coarse	64	81	96	98·3	82·7	96·6
	Fine	55	79	95	97·6	86·8	97·0
Barnsley Y.M.	Coarse	15	41	91	97·7	80·6	97·5
	Fine	13	35	91	97·9	74·9	98·1
Barnsley H.M.	Coarse	40	61	86	94·1	73·0	94·2
	Fine	40	63	89	94·4	75·0	95·8
60% Barnsley H.M. 40% Barnsley Y.M.	Coarse Coarse	45	66	87	93·5	75·3	95·4
60% Barnsley H.M. 40% Barnsley Y.M.	Fine Coarse	23	52	89	95·9	79·9	96·6
60% Barnsley H.M. 40% Barnsley Y.M.	Fine Fine	19	45	89	96·7	77·9	97·2

The "coarse" grinding in these tests was approximately 90 per cent. through ¼-in. with 60 per cent. through ⅛-in.; and the "fine" grinding was 100 per cent. through ¼-in. with 85 per cent. through ⅛-in.

The Effect of Blending.—From observations on the effect of the quality of the coal on the size of the coke yielded, it would be expected that the blending of a poor with a good coking-coal would increase the size, and this is generally so. For example, Figs. 2, 3 and 4 show the effects of (1) adding increasing quantities of a good coking coal, Parkgate M, to a non-coking coal, Barnsley B; (2) adding fusain to these Parkgate-Barnsley blends; and (3) adding strongly-coking mixtures to a weakly-coking Derbyshire coal. All the tests were made in the experimental oven with a coking-time of 18 to 19 hours.

With the Parkgate-Barnsley blends, there was a regular increase in the size of the coke as the proportion of Parkgate was increased up to 70 per cent., and the addition of fusain further increased the size. With the weakly-coking Derbyshire coal, the effect on the size of the coke of blending with a good coking coal was marked.

The Effect of the Method of Charging the Oven.—Sufficient data have been recorded in our first report (*see* p. 25) to show that, in general, the top-charging of an oven causes the coke to contain a rather greater proportion of the larger sizes than when the coal is cake-charged, but that the amount of breeze is slightly greater.

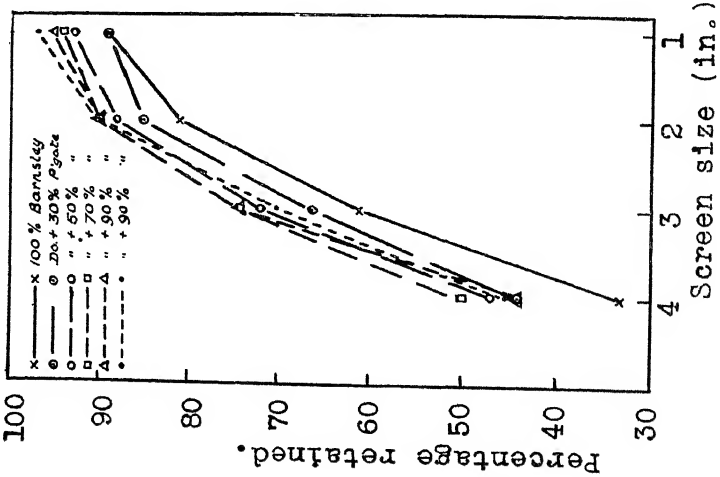


FIG. 2.—The Effect on the Screen Size of Coke of Blending Parkgate M with Barnsley B Coal in Different Proportions.

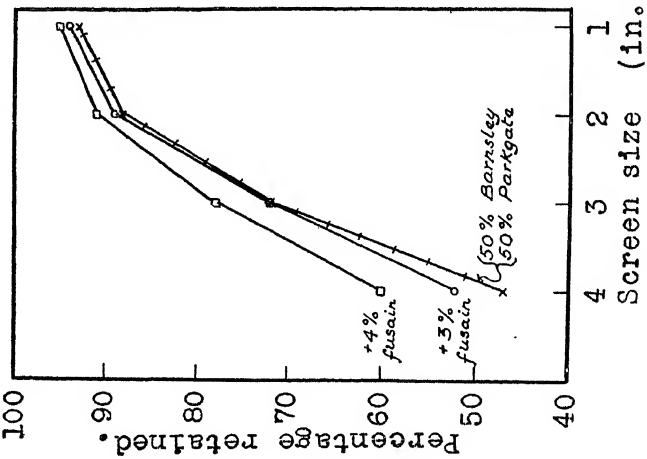


FIG. 3.—The Effect on the Screen Size of Coke of Adding Fusain to a Blend of 50 per cent. Parkgate M 50 per cent. Barnsley B.

The Effect of the Rate of Heating.—The rate of heating the charge, as determined by the width of the oven and the flue temperature, greatly affects the size of the coke produced from a given coal, the coke being smaller the faster the rate. It thus

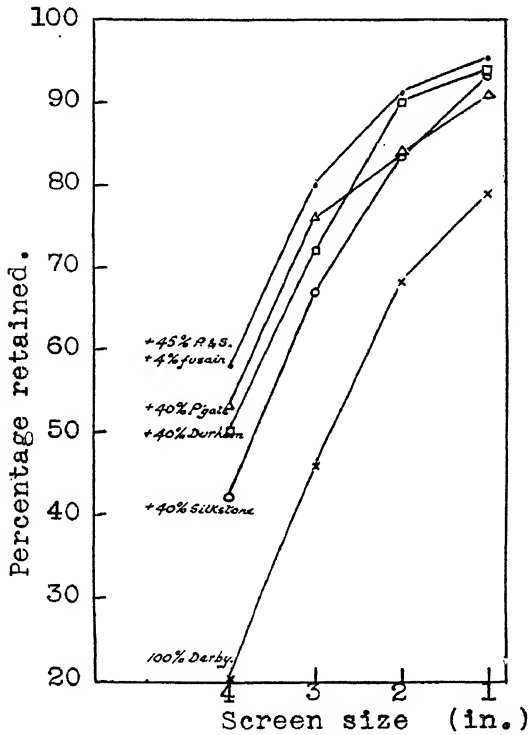


FIG. 4.—The Effect on the Screen Size of Coke of Adding Strongly-Coking Coals to a Derbyshire Coking Slack.

results that the coke made in modern narrow ovens with high flue-temperatures is much smaller than that formerly made.

The effect of the rate of heating is illustrated in Fig. 5, which records the size of the coke discharged from the experimental oven (18 in. in width) when the coking-time for three different slacks ranged from 13 to 26 hours. The effect is greatest on the proportions of 4-in. and 3-in. size and varies with the coal.

Although for the fastest rate of heating in the experimental oven the amount of 4-in. coke, as recorded in Fig. 5, was small, it is considerably greater than is usual in practice, the reason being that the coking-time was shorter than for a full-scale oven of the

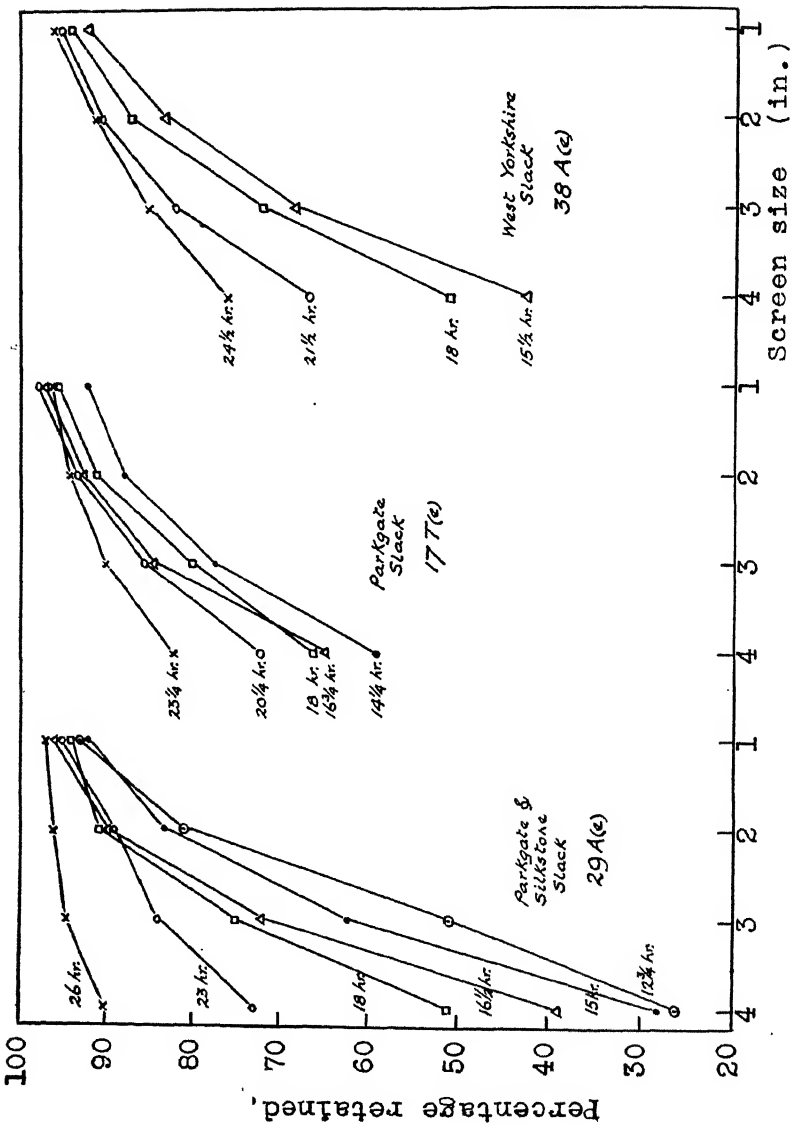


FIG. 5.—The Effect on the Screen Size of Cokes of Varying the Coking Time in an 18-in. Oven. Three Coals.

same width, owing to the lower bulk-density of the charge. The fact that so little 4-in. coke is produced from modern ovens does not appear to be of much moment so far as blast-furnace coke is concerned. Of more significance is the fact that, with the most rapid rates of heating, the amounts of 2-in. and 1-in. coke are reduced; for it is desirable to produce, as blast-furnace coke, as big a yield as possible of coke screened at the ovens, over, say, 1 in.

The Size of Coke at the Blast-Furnace

The coke discharged from an oven can suffer considerable reduction in size before it reaches the blast-furnace. It is important to realise that every fall which the coke experiences will cause its breakage. Although this may not matter, or may be advantageous, with respect to pieces over 3 in. in size, the simultaneous formation of breeze is decidedly disadvantageous for, unless the breeze is removed, its presence may interfere seriously with the performance of the blast-furnace.

The Effect of Handling.—Two examples of the effect on the size of coke of its handling between the ovens and the blast furnaces can be given.

Table IV.—The Handling of Coke.

	(a) Transport by Rail	Slide (ft.)	Fall (ft.)
(i)	From screens by loading booms to wagons	14	5
(ii)	Transport by wagons, 2 miles	nil	nil
(iii)	Wagon weighed and coke dropped to bunkers	nil	10
(iv)	From bunkers to screens and skip	18	6
(v)	Skip elevated and coke dropped on to upper bell	nil	20
(vi)	From upper bell to lower bell		
(vii)	From lower bell to stock level	nil	6
	Total	32	47
	(b) Transport by Aerial Ropeway.		
(i)	From screens to shoot	nil	1
(ii)	From shoot to belt	8	nil
(iii)	From belt to (full) hopper	nil	4
(iv)	From hopper to ropeway bucket	15	3
(v)	Transport by ropeway, 2 miles	nil	nil
(vi)	From ropeway bucket to inclined shoot	nil	10
(vii)	From shoot to hopper	5	nil
(viii)	From hopper to wagon	8	5
(ix to xiii)	As (iii) to (vii) for transport by rail	18	42
	Total	54	65

At one plant coke could be delivered to the blast-furnaces either by railway or by aerial ropeway (see *J. Iron and Steel Inst.*, 1933,

1, 93). The treatment to which it was subjected between the coke-oven screens and the stock-level of the furnace can be tabulated as in Table IV.

The most severe fall was from the skips to the stock-level and, whilst the effect of the handling up to the skips could be discounted by screening the coke, any breeze formed subsequently passed into the furnace.

An attempt was made to determine the effect on the size of the coke of this handling by simulating it in the laboratory. A sack containing 50 lb. of over 2-in. coke was dropped on to a bed of coke from the appropriate height, the fall being preceded by a slide when that had occurred in practice. After each treatment the coke was screened. The result is shown diagrammatically in Fig. 6. Curve No. 1 represents treatments i and iii of Table IV (*b*), whilst curves Nos. 2 to 8 relate to the treatments iv, vi, viii, ix, x (xi and xii) and xiii. The "experimental" handling of the coke was more severe than in practice by reason of the screening introduced between each treatment. The average size-analysis of the coke delivered at the top of the furnace screens by aerial ropeway was determined, and can be compared with the size of the test coke after equivalent treatment (curve No. 5 of Fig. 6):

Size of screen, in.: ..	4	3	2	1½	1	½
Average size of coke at furnace screens, per cent. on:	2·4	11·8	52·9	77·8	91·8	96·9
Average size of test coke, per cent. on:	5·7	19·3	55·0	70·8	86·1	94·9

Rather more breakage occurred during the laboratory treatment, as regards the 1½-in. and smaller sizes, than in practice.

At another plant, the handling of the coke was as summarised in Table V.

TABLE V.—DETAILS OF HANDLING BETWEEN COKE-OVENS AND BLAST-FURNACES.

	Drop (ft.)	Cumulative (ft.)
(i) From cross belt from coke-ovens to furnace bunker belt No. 4	7½	7½
(ii) From bunker belt No. 4 to bunker belt No. 3	9	16½
(iii) From bunker belt No. 3 to bunker belt No. 2	9	25½
(iv) From bunker belt No. 2 to bunker belt No. 1	9	34½
(v) From bunker belt No. 1 to bunker slide No. 1	4	38½
(vi) From bunker slides Nos. 1, 2, 3, and 4 to bunkers 1, 2, 3 and 4	10 to 20	
Total height of drop to bottom of bunker No. 4		
Total height of drop to bottom of bunker No. 1		48½*

* Assuming minimum drop of 10-ft. to bunker.

The size of the coke at different points in the system outlined in Table V was determined, with the results given in Table VI.

TABLE VI.—SIZE OF COKE AT DIFFERENT POINTS OF SYSTEM IN TABLE V.

Per cent. of Coke on :	4-in.	3-in.	2-in.	1½-in.	1-in.	½-in.
At cross belt . .	3·8	19·2	63·7	81·7	94·1	99·1
At bunker belt No. 4 .	2·8	16·9	63·6	81·8	94·1	98·1
At bunker belt No. 3 .	1·8	13·2	57·9	78·1	92·5	98·4
At bunker belt No. 2 .	1·1	10·8	53·6	76·4	91·8	98·4
At bunker belt No. 1 .	0·5	6·8	48·5	73·8	91·2	98·2
In No. 4 bunker . .	nil	4·2	35·8	64·4	85·7	96·0
In No. 1 bunker . .	nil	1·9	30·8	60·9	84·6	95·5

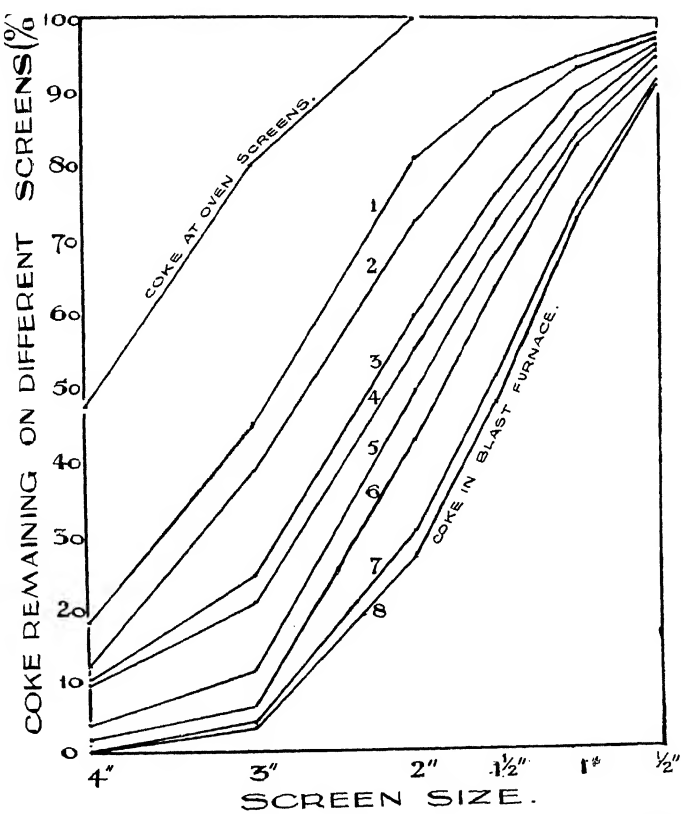


FIG. —The Effect, on the Screen Size of Coke, of each Drop during Handling between the Coke Ovens and Blast Furnace.

The size-analyses of the samples from the belts are averages of 12 daily samples, each of 100 lb., taken when the belts were stationary. The bunker samples were collective belt-samples totalling 10 cwt. dropped into a cleared bunker. This coke had a $1\frac{1}{2}$-in. shatter index of about 74.

In practice, the breakage of coke on falling from a given height is less than would occur with a "shatter" test apparatus operated to provide the same total height, because the fall is usually on to a bed of coke instead of on to a steel plate, as in the shatter test, and because in a continuously falling stream of coke some support is afforded to any given portion of the stream. The effect of a fall on to coke as compared with a fall on to a steel plate is illustrated by tests in which the base plate of a shatter test apparatus was covered by a layer of 1–$1\frac{1}{2}$-in. coke, and the test carried out otherwise in the usual manner, 50 lb. of over 2-in. coke being dropped four times from a height of 6 ft. The average of three such tests gave the following result:

		Shatter Indices.	
		2-in.	$1\frac{1}{2}$-in.
Dropping on to steel plate	..	87·9	93·9
Dropping on to layer of coke	..	95·8	97·5

Even after making due allowance for the fact that during the normal handling of coke it does not fall directly on to metal, it is not easy to forecast, from the results of shatter tests, the amount of breakage that will occur in practice; for, although more breakage occurs in proportion to the amount of handling the coke receives, there is not a progressive increase in the amount of through $1\frac{1}{2}$-in. size formed, because there is progressively less over $1\frac{1}{2}$-in. size coke to be broken. Moreover, the early breakage of coke occurs along the major lines of weakness of the pieces; thereafter, the pieces to be broken are "harder." Some tests with a Yorkshire coke illustrate these effects. In shatter tests, the coke was allowed to drop 12 times from a height of 6 ft. and screening analyses were made after each drop. In Fig. 7 the average results of three such tests are shown by expressing the amounts of undersize at $1\frac{1}{2}$-in., 1-in. and $\frac{1}{2}$-in. in the coke after each drop as percentages of the amounts of oversize present at these screens before the drop. With few exceptions, the values fall on straight lines, indicating that the coke became "harder" on repeated dropping, so that proportionately less breakage occurred. It is of interest that the amount of $\frac{1}{2}$-in. coke recorded throughout the test should be constant, for this is an indication that the formation of $\frac{1}{2}$-in. coke is not a measure of its "hardness;" we have already shown that the $\frac{1}{2}$-in. index records, rather, the "abradability" of the coke.

In Fig. 8 the results of similar tests for four different cokes, as regards the $1\frac{1}{2}$-in. size, are recorded. These results show that, over the normal range of blast-furnace cokes of the Midland area

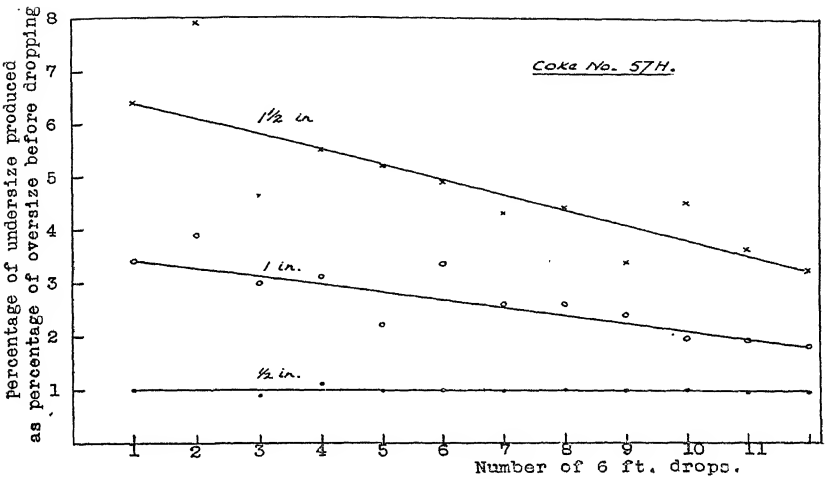


FIG. 7.—The Increase in Impact Hardness of Coke on Handling shown by Decrease in Percentage Undersize Produced in Successive 6-ft. Drops.

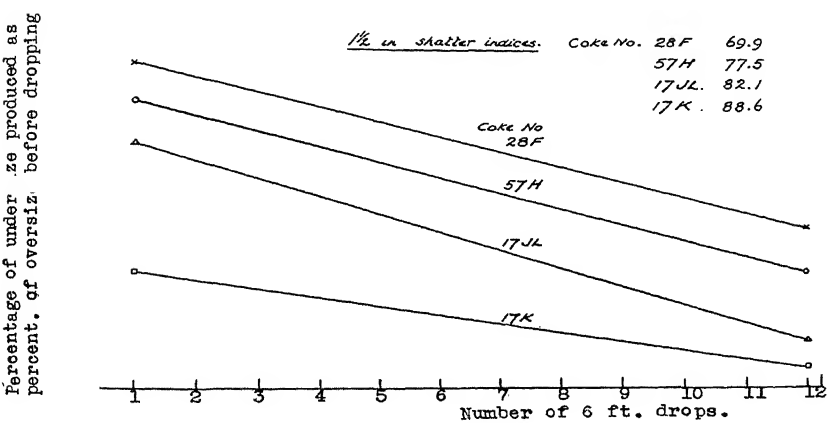


FIG. 8.—The Increase in Impact Hardness on Handling is Proportional to the Original Shatter Index of Midland Cokes of Average Hardness.

having $1\frac{1}{2}$-in. shatter indices between 70 and 82, the amount of breakage on dropping varies with the shatter index, irrespective of the number of drops.

The Size of Coke for Cupolas.—The Northern Coke Research Committee, in collaboration with the British Cast Iron Research Association, have compared the behaviour, in a 5-ft. cupola melting about 110 tons of pig-iron and heavy scrap-iron in an 8-hour day, of the same quality of beehive coke when ungraded or graded 3-$1\frac{1}{2}$-in. and 2-1-in. sizes in successive trials (Briscoe, *Foundry Trade Journal*, 1929, **40**, 307). With the smallest size of coke (which was successfully used in a smaller cupola) the cupola began to work sluggishly and the test had to be suspended. The principal results for each of the other trials are recorded in Table VII.

TABLE VII.—COMPARISON OF UNGRADED AND GRADED COKE IN A CUPOLA.

Size of Coke	Ungraded (93% over 2-in.)	Graded (94% 3-$1\frac{1}{2}$-in.)
Melting ratio, metal/coke	13·0	12·6
Metal production, tons per hr.	13·9	13·26
Blast volume, cu. ft. per hr.	305,000	272,000
Blast pressure, inch W.G.	16·00	16·34
Metal temperature at spout, °C.	1334	1349
Metal temperature at ladle, °C.	1288	1303
Top-gas analysis : (*a*) at wall, CO_2	12·6	10·3
CO	7·1	9·5
O_2	0·6	2·5
(*b*) at centre, CO_2	7·8	8·2
CO	3·6	6·9
O_2	8·1	5·4
Top-gas temperature °C., at wall	217	147
at centre	113	66

Judged by the foundryman's criteria of metal/coke ratio and rate of melting, the ungraded coke was superior to the graded. A noticeable feature of the trial with the graded coke was the low temperature of the top-gas and its uniform analysis. The method of charging the ungraded coke caused a loose pack to form down the middle of the cupola. This, and the greater voids in the charge as compared with a charge of graded coke, allowed more air to be fed at the same blast-pressure so that combustion was more nearly complete and the temperature higher.

The ungraded coke contained about 33 per cent. of over 4-in. size, and it is possible that this gave it its superiority. Had the graded coke been of 4-2-in. or even of 3-2-in. size, the more uniform condition of the fuel-bed in the cupola obtainable by such grading might have been advantageous.

The Size of Coke for Stoves and Boilers.—In slow combustion stoves in particular, where disturbance of the fuel-bed as combustion proceeds is slight, it is important that the size of the coke should be chosen in relation to the size of the stove. The size of the coke must be such that the bed will settle uniformly without arching, a performance most readily obtainable when the size is closely graded and the pieces are cubical.

Cubical pieces are obtained from cokes for which the 2-in. and $1\frac{1}{2}$-in. shatter indices bear a normal ratio ,*e.g.* 61/75; 67·5/80, 74/85. When there is a departure from the normal, which can be rectified by suitable blending, *e.g.* with coke dust, it is found that the coke is less "blocky" and is of irregular shape. Cokes of low shatter index are usually prismatic or "fingery" and yield prismatic pieces when "cut" in the preparation of coke nuts. Hardness as well as blockiness is desirable in a coke for the production of nuts of regular cubical shape.

The sizes of gas coke recommended by the London and Counties Coke Association for different purposes are as follow:

Grade	Size	Use
No. 1 Large or unbroken	Over 2-in.	Very large boilers and steam raising.
No. 2 Broken	Within the limits 1 to 2-in.	Open grates, large central-heating boilers.
No. 3 Coke boiler nuts	Within the limits $\frac{1}{2}$ to $1\frac{1}{4}$-in.	Small central-heating boilers, large domestic boilers.
No. 4 Forge beans	Within the limits $\frac{3}{8}$ to $\frac{5}{8}$-in.	Small domestic boilers, closed stoves, kitchen ranges, mechanised stoves.

The limits are square mesh screens and intermediate sizes within the limits are permissible.

No standardisation has as yet been attempted of the grades of sized cokes prepared from by-product oven coke, and there is considerable variation in the products marketed in different districts or at different plants in the same district. In any standardisation that might be contemplated due regard would have to be paid not only to the sizes of the screens, but to their type, *e.g.* whether round or square hole, and their screening efficiency. Examples of the grades of sized coke marketed in the Midland area are as follow:

From a Yorkshire Plant.

(Round hole screens.)

No. 1 ("apples")	$2\frac{1}{2}$–$1\frac{1}{2}$-in.
No. 2 ("walnuts")	$1\frac{1}{2}$–1-in.
No. 3	1 –$\frac{1}{2}$-in.

From a Derbyshire Plant.

(Round hole screens.)

No. 1	$2\frac{1}{2}$–2-in.
No. 2	2 –$1\frac{1}{4}$-in.
No. 3	$1\frac{1}{4}$–$\frac{5}{8}$-in.
No. 4	$\frac{5}{8}$–$\frac{1}{4}$-in.

The No. 1 size from the Yorkshire plant is used successfully in all sizes of central-heating boilers down to 1 ft. square grate area; the No. 2 size from the same plant is used in domestic hot-water boilers of which the firepot area may be only 8 by 8-in., and the No. 3 size is used in slow-combustion stoves and for forge fires.

From a study of the behaviour of sized coke on combustion in stoves, using ½-in. grades hand-screened through square holes, the following generalities may be stated:

(1) The bulk-density of the charge, for a given brand of coke, increases with decrease in the size below 1½-in., and the resistance to draught increases correspondingly.

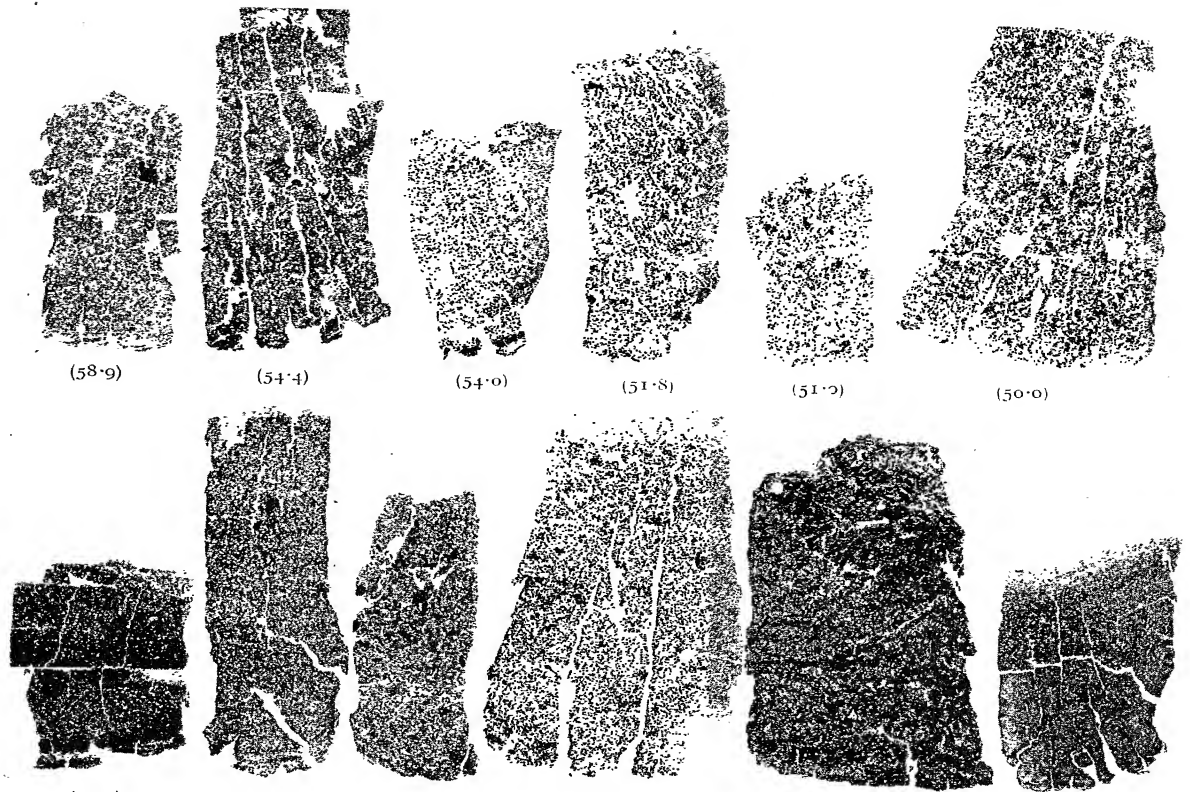
(2) Sizes less than 1½-in. are considerably more reactive, *i.e.* present a smaller oxidation zone, than the larger sizes.

(3) Sizes less than 1½-in., when burnt in a fuel-bed 12 in. deep, reduce carbon dioxide to monoxide more rapidly than do the larger sizes.

(4) Sizes less than 1½-in. produce a lower average fuel-bed temperature than the larger sizes, and there is a marked lowering of the temperature if the size is less than 1-in.

(5) The liability to form troublesome clinker increases with decrease in the size of the coke.

Oven coke 1½–1-in. (round hole) grade is particularly useful for small domestic hot-water boilers, such as are relighted daily, for a bed of coke of this size settles readily in the smallest boiler, attains a high temperature (when the chimney-draught is adequate) and, if not overloaded, is not liable to give trouble through the formation of clinker. On the other hand, it may be difficult to maintain a fire overnight with this size of coke, if it is of low specific reactivity, unless a special feeding hopper is used. The 1–½-in. grade of coke is the most useful for slow-combustion stoves, where the low average temperature of the fuel-bed is not a disadvantage. For central-heating boilers of comparatively large size the 2½–1½-in. grade is most serviceable, since it is less liable than the smaller sizes to form clinker.



Fi parison of Large (above) and Small-pored (below) Cokes of the same Total Porosity.

CHAPTER VI

THE STRUCTURE OF COKE

THE macro-structure of by-product coke was considered in some detail in "Coke for Blast-furnaces" (Part I, Chapter III), the shape and manner of the fracture of the pieces of coke being described in relation to the quality of the coal from which the coke was made and the conditions of its carbonisation. Several examples of the micro-structure of cokes were also given. A further study of the micro-structure of cokes has not proved profitable as far as commercial by-product cokes are concerned, but useful information has been so obtained in laboratory researches regarding the manner of formation of coke, and is recorded in the description of these researches (*see* p. 271)

In a study of "Rose" sections of cokes (*i.e.* polished sections with the pores infilled with plaster-of-paris so as to be readily visible), it is observed that, in the harder cokes made from Durham and Welsh coals, the average size of the pores is less than in weaker Yorkshire cokes made under the same conditions as regards the method of charging and the rate of heating. This is illustrated by Fig. 9, on which longitudinal sections of six pairs of cokes are shown, each pair having been chosen with about the same total volume of pores (recorded in brackets). Each of the sections in the upper row has pores of larger size than in the corresponding section in the lower row, and therefore has a lighter general appearance. Since, in each pair, the total volume of pores (recorded in Fig. 9) was about the same, the pore-surface of the darker-looking sections must be greater. For example, with two cokes each having 50 per cent. of pore-space, but with pores of average diameters 1 mm. and $\frac{1}{2}$ mm., respectively, the numbers of pores would be in the ratio 1 to 8 and their surfaces in the ratio 1 to 2.

The small-pored cokes have the more regular structures and, when they are "cracked" to produce nut sizes, do not make much dust. Fig. 9 shows that a small-pored coke has fewer fractures than the corresponding large-pored coke, and it can be said that, in general, small pores in a coke are desirable.

CHAPTER VII

THE SAMPLING AND ANALYSIS OF COKE

THE quality of coke supplied has often been the subject of dispute between buyer and seller. Had standard methods of sampling and analysis of the consignments been available, many of the disputes need not have arisen and most of them could have been amicably settled. In the absence of recognised standards, buyer and seller have been forced to use methods for the sampling and analysis of coke which, though they may have come to be regarded as fairly satisfactory through familiarity in their use, have been primarily devised for dealing with consignments of coal or ore. It can be understood that with methods of sampling not necessarily applicable to coke, and with methods of analysis of uncertain degree of accuracy, an unfortunate lack of trust in each other's results may have been engendered as between buyer and seller.

The results of tests made to determine the degree of error likely in the sampling and analysis of coke by specified methods are here recorded, and it is shown how error can be minimised.

THE SAMPLING OF COKE

The work of E. S. Grumell and A. C. Dunningham (*British Standard No.* 403) has provided a rational basis for the sampling of coal. In that work, the "variability" of coal in a consignment, that is to say, the relative difficulty of sampling it, was found to increase with the ash-content of the coal. This greater variability of high-ash coals could be discounted by increasing the number of increments and the gross weight of the sample taken. The variability of a consignment of a material is expressed by the average error, or the average deviation from the mean value, of a large number of determinations on an ingredient of the consignment, *e.g.* of the ash of coal. For deliveries of small coal under 1-in. in size and of 10 per cent. ash-content, a degree of accuracy such that in 99 out of 100 tests the ash-content would be correct within 1 per cent. can be obtained if gross samples of 46 lb. are taken by 23 increments of 2 lb. each (*see* Grumell and Dunningham, *loc. cit.*, p. 41).

It is important to state clearly the significance of this expression of the degree of accuracy to be expected. It means that, supposing

the true ash-content of the coal to be 10 per cent., once only in 100 tests would the ash-content of the sample taken be found to be less than 9 or more than 11 per cent. In the remaining 99 tests, a closer approximation to the true ash-content could be expected; in 82 of them the value found would be within 0·5 per cent., and in 50 of them it would be found to be within 0·26 per cent. of the true ash-content.

When dealing with the sampling of coke, it can be assumed that there will be a lower average error than for coal so far as the ash-content is concerned; for coke is usually made from small coal that has been cleaned to a fairly constant ash-content and crushed. The size of pieces of dirt mainly responsible for high ash-content is therefore small in relation to the size of the coke. The average error is lower in consequence, and there is greater ease of sampling than with coal.

For moisture there is greater variability than for ash in coke. The same method of sampling therefore results in a greater average error in the moisture-content than in the ash-content of the coke. This is because, during the quenching of coke, the smaller sizes offer relatively greater surfaces for the retention of moisture than do the larger pieces. The average moisture-content of a consignment of coke is therefore affected by its size when quenched. For this reason, the British Standard Specification for the Sampling and Analysis of Coke (*No.* 496) differentiates between two classes: Class I.—Cokes containing not more than 10 per cent. of through 2-in. pieces at the place of manufacture (*i.e.* when quenched), and graded coke even if less than 2 in. in size; Class II.—All other varieties of cokes.

The experimental work upon which the recommendations of the British Standards Institution in their Specification for the Sampling and Analysis of Coke were based is here recorded, so that their necessity can be judged, and practical methods of giving effect to them are outlined.

An Experimental Study of the Sampling of Metallurgical Coke.—We have made an experimental study of the "variability" of metallurgical (by-product oven) coke, taking the moisture-content as being the constituent most likely to show variation.

Quenching by Water-spray.—The coke from an oven was quenched in a coke-car by passing it under a horizontal spray-pipe. The average moisture-content of the different charges of the coke was varied by altering the number of times the car passed beneath the spray-pipe and the rapidity of its passage.

The car was built up lengthwise from 12 plates which conveniently indicated zones from which to take separate samples. By dividing the width of the car, by eye, into 4 equal parts, 48 zones were provided. From each zone, three samples ("increments") about 5, 2½ and 1-lb. in weight, were collected in weighed

and numbered tins. The 5-lb. and 2½-lb. samples were made up of unbroken pieces but to obtain each 1-lb. sample it was usually necessary to break a larger piece of coke.

As soon as the sampling was completed, the tins, 144 in number, were sealed by lever lids and weighed. The samples were then taken to the laboratory and dried in batches of 25 or 50, the lids of the tins having been removed. The drying-oven consisted of a steel hot-plate, 6 ft. by 3 ft., covered with asbestos sheet and enclosed by steel sheets. Luminous gas-jets (*i.e.* without primary aeration) from three parallel burner-tubes extending the full length of the gas hot-plate provided a temperature of about 160° C., and drying was completed in 3½ to 8 hours.

The principal results of the tests are summarised in Table VIII.

TABLE VIII.—SUMMARY OF TESTS ON SAMPLING COKE QUENCHED BY WATER-SPRAY.

Test No.	1	2			3		
Weight of increments, lb.	1	5	2½	1	5	2½	1
Mean moisture content of 48 increments—%	0·70	3·0	3·65	4·25	6·15	6·80	8·0
Maximum moisture content—%	3·85	10·5	11·40	14·80	15·80	15·80	22·50
Minimum moisture content—%	nil	0·1	0·25	0·40	0·55	nil	0·25
Average deviation from mean	0·45	1·8	2·45	2·85	3·30	3·05	4·30

Test No.	4			5		
Weight of increment, lb.	5	2½	1	5	2½	1
Mean moisture-content of 48 increments—%	9·50	10·32	10·60	14·05	15·7	15·35
Maximum moisture content	17·00	22·50	24·80	25·80	23·5	22·80
Minimum moisture content	1·60	2·4	1·20	3·60	4·7	4·55
Average deviation from mean	3·15	3·65	4·40	3·45	3·5	4·70

Except in Test No. 5, the mean moisture-content of the samples (which represent the "increments" of a gross sample) increased with decrease in their weight. No doubt, when collecting a 1-lb. increment, the sampler would take either a small piece, under 2 lb. in weight which would conveniently yield a 1-lb. piece when broken, or, when breaking off a 1-lb. piece from a larger lump, would include overmuch of the outer layer, which contains excess of moisture. When collecting a 5-lb. increment, the sampler would take either two pieces each weighing about 2½ lb. or, occasionally, a single lump of coke.

For a true average sample to be obtained, the weight of the increment taken when sampling a consignment of coke should

approximate to the average weight of the unbroken pieces in the consignment. This, in our experience, is from 2 to 3 lb. The average deviations from the mean values for moisture-contents given in Table VIII are nearly the same for the 5-lb. and 2½-lb. pieces.

The results recorded in Table VIII are shown diagrammatically in Fig. 10. It is evident from the graphs that, so far as the 5-lb. and 2½-lb. increments are concerned, the average deviation from the mean value for moisture-content, which gives an index of

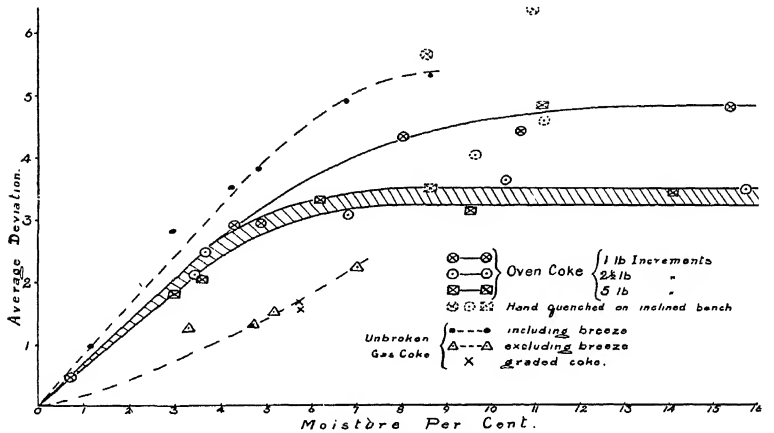


FIG. 10.—The "Average Deviations" in Sampling Cokes of Different Moisture Contents.

the "variability" of the consignment of coke sampled with respect to the moisture it contained, increased rapidly with the mean up to about 5 per cent. but did not increase much thereafter. This is because there is a maximum amount of moisture that a particular kind of coke, quenched in a particular way, can absorb. The coke used for these tests was a South Yorkshire coke of good quality made from top-charged coal coked in a 20½-in. oven in 30 hours. For the normal quenching of this coke, the car is passed twice (once quickly and once slowly) beneath a horizontal water-spray, the coke being in a thin layer in the car. This method of quenching affords greater ease of control than any other and, as a result, the moisture-content of the coke leaving the plant is always low.

Central Quenching.—The usual method of quenching at a modern battery of coke-ovens is central quenching. This method was studied at a South Yorkshire plant, where the coal is carbonised in 14-in. ovens in about 12 hours. The coke upon which the tests were made was pushed on to a car, holding 8 tons, which, whilst

stationary, was treated with 4,000 gallons of water discharged in 35 seconds from an overhead tank through two 9-in. pipes each having five rows of $\frac{3}{8}$-in. holes at 1-in. centres. After having drained, the coke was spread out on a wharf to "steam off" and 20 to 30 minutes later was loaded on to a conveyor.

The coke was sampled from the moving conveyor at different points across its width. The same number and character of samples ("increments") was taken as in the test of quenching by water-spray, and the moisture-contents were determined in the same manner. The principal results are summarised in Table IX:

TABLE IX.—SUMMARY OF TESTS ON SAMPLING OF COKE, QUENCHED CENTRALLY.

Weight of Increments, lb.			
Mean moisture content of 48 increments, %	3·6	3·45	4·85
Maximum moisture-content, %	11·15	8·75	14·4
Minimum moisture-content, %	0·4	nil	0·6
Average deviation from mean	2·05	2·1	2·9

As before, the mean moisture-content and the average deviation from the mean are higher for the 1-lb. than for either the $2\frac{1}{2}$-lb. or the 5-lb. samples. The average deviations from the mean values are shown in Fig. 10.

Hand-hose Quenching on an Inclined Bench.—Tests on coke quenched by hand-hose were made at a South Yorkshire plant where a charge of about 6 tons is pushed through a guide on to an inclined bench and there quenched. Water-sprays within the guide were not used because the oven-wall was of silica which is liable to spall if sprayed.

Since the coke is not evenly distributed on an inclined bench, uniform quenching is difficult. Firing of the coke after hand-quenching is not uncommon and may necessitate "spot" quenching, with consequent excess of water locally. Moreover, when a number of ovens is being pushed, the "fans" of coke from adjacent ovens may overlap at the lower part of the bench so that drainage of excess water is impeded.

For the test, a time was chosen when twelve wagons of coke (120 tons, representative of about 20 ovens) were standing. Samples, each comprising, 5, $2\frac{1}{2}$ and 1-lb. increments, were taken from each wagon at points one-sixth of diagonal lines drawn from each corner and at 1 ft. depth. The average deviations from the mean moisture-contents were higher than in the tests on cokes quenched either by water-spray or centrally. A repeat test was therefore made. The principal results of both tests are summarised in Table X.

TABLE X.—SUMMARY OF TESTS ON SAMPLING OF COKE, QUENCHED BY HAND-HOSE.

Test No.	A			B		
Weight of increment, lb.	5	2½	1	5	2½	1
Mean moisture content of 48 increments, %	8·6	9·6	10·9	10·1	11·15	8·5
Maximum moisture-content, %	19·2	21·3	32·1	24·7	28·2	30·6
Minimum moisture-content, %	2·1	1·6	0·5	0·5	1·9	0·4
Average deviation from mean	3·5	4·05	6·4	4·85	4·6	5·65

If it be assumed that the average deviations from the mean moisture-contents of the coke samples (of about 10 per cent. moisture-content) quenched by water-spray were "normal," the departure from the normal for these hand-quenched cokes can be indicated thus :

Weight of Increments (lb.)		2½	
Mean moisture-content—			
Test A	8·6	9·6	10·9
Test B	10·1	11·15	8·5
Average deviation from mean—			
Test A	3·5	4·05	6·4
Test B	4·85	4·6	5·65
"Normal" average deviation from mean (Fig. 10)	3·3	3·5	4·5

From these tests it is evident that, when coke is hand-quenched on an inclined bench, the "variability" of samples as regards moisture-content may be markedly greater than when the coke is quenched in a thin layer on a coke car. Unfortunately, the variability of hand-quenched cokes cannot be expressed in a simple graphical form as for cokes quenched by water spray. If, however, the marked variability is associated with a high average moisture-content, as is likely, such cokes would fall in the Class 2 provided by British Standard Specification for the Sampling and Analysis of Coke, irrespective of their size.

Tables recording details of all the tests made are given in an appendix to this chapter (p. 62).

It is clear from these results that a reliable sample of coke for the determination of moisture-content can often only be obtained if a large number of increments, representing the coke from different portions of the consignment, is taken. The "variability" of the sample as regards moisture content is indicated by the average deviation from the mean value in a series of about 50 tests. The greater the average deviation, the larger the number of increments required to provide a sample of the desired accuracy. The number of increments necessary to ensure that in 99 tests out of 100 the

sample enables the moisture-content to be determined within 1 per cent. of its true value can be calculated in accordance with the theory of errors, an approximate solution being given by the formula:

$$n = \left[\frac{0{\cdot}85E}{0{\cdot}255}\right]^2 = 11E^2$$

in which n is the number of increments required and E is the average deviation from the mean value.

From Fig. 10, n can be determined for cokes of different moisture-contents. For 2½-lb. increments the values are:

Moisture-content of coke, %	Number of Increments (n)	Minimum Weight of Gross Sample, lb.
Up to 2	20	50
Over 2 and under 3	44	110
Over 3 and under 4	74	185
Over 4 and under 5	95	238
Over 5 and under 6	112	280
Over 6 and under 7	118	295
Over 7 and under 8	126	315
Over 8	134	335

These values relate in particular to Yorkshire cokes quenched on a coke-car, but it is reasonable to assume, on our present knowledge, that the results recorded in Fig. 10 and the values for the numbers of increments based on them, should apply to all oven cokes, in whatever way quenched, provided that the moisture-content does not exceed 5 per cent. For cokes containing more than 5 per cent. of moisture, a "factor of safety" should be introduced.

These are, in effect, the conclusions drawn by the British Standards Institution. For their Specification No. 496, 1933, they have adopted the values for increments calculated from Fig. 10 for cokes containing 5 per cent. of moisture or less, with not more than 10 per cent. of through 2-in. size at the place of manufacture. If the coke contains more than 5 per cent. of moisture it is placed in Class 2, and the number of increments required to be taken to secure a "standard" sample must be augmented.

Fig. 10 shows that, for coke quenched by water-spray in a coke-car and containing less than 5 per cent. of moisture, but little greater average deviation from the mean value was obtained when the increment weighed 1 lb. instead of 2½ lb. The taking of 2½-lb. increments, however, provides a necessary "factor of safety." Other "factors of safety" are provided in the schedule of minimum weights of gross samples of different varieties of cokes recommended in the British Standard Specification for the Sampling and Analysis of Coke (*loc. cit.*, p. 7). This is reproduced in Table XI.

TABLE XI.—MINIMUM WEIGHTS OF GROSS SAMPLE.

Moisture-Content per cent.	Class No. of Increments	Class Minimum Weight of Gross Sample, lb.	Class 2† No. of Increments	Class 2† Minimum Weight of Gross Sample, lb.
Up to 2 . .	20	50	35	
Over 2 and up to 3	44	110	70	175
Over 3 and up to 4	74	185	120	300
Over 4 and up to 5	95	238	170	425
Over 5 and up to 6	112	280*	235	588
Over 6 and up to 7	118	295*	280	700
Over 7 and up to 8	126	315*	300	750
Over 8 . .	134	335*	305	763

* Graded coke only.
† The numbers of increments required for cokes of Class 2 were based on comprehensive tests carried out by the South Metropolitan Gas Company.

This study of the variability of the moisture-content of different cokes has been extended by E. S. Grumell (*Trans. Inst. Min. Eng.*, 1933, **86**, 126), who sampled large consignments received under contract by Imperial Chemical Industries, Ltd. The average deviations from the mean moisture-content, representing the average errors, are recorded in Table XII.

TABLE XII.—AVERAGE ERROR IN MOISTURE-TESTS, OBTAINED BY SAMPLING WAGONS.

Coke*	No. of Wagons	Average Moisture %	Average Error	Maximum Moisture %	Minimum Moisture %
D1 .	96	2·43	1·63	7·60	0·19
Y2 .	100	2·77	1·43	7·87	0·16
Y3 .	94	2·78	1·64	7·15	0·16
Y4 .	100	2·82	1·42	10·48	0·20
Lancs. 5	100	4·70	1·98	13·12	0·33
L6 .	62	5·36	1·30	10·40	2·60
Y7 .	50	5·80	2·46	15·57	0·55
Y8 .	34	5·87	1·60	10·59	1·14
Y9 .	33	7·15	3·01	15·51	1·02
N.S.10 .	100	7·21	2·08	12·50	2·00
L11 .	54	9·75	3·09	18·00	3·35

* D, Durham ; Y, Yorkshire ; L, Lancs. ; and N.S., North Staffs.

The average errors of all these cokes lie within the curve for 5-lb. increments in Fig. 10, and eight out of the eleven values lie nearly on the curve.

The Collection of a Gross Sample of Coke.—The recommendation of the British Standard Specification as to the number of increments, each not less than 2½ lb. in weight, that should be taken to obtain a satisfactory gross sample of a coke, relates to either a single consignment or to a number of consignments of the same quality of coke delivered over a period. It would be difficult to collect the requisite number of increments from a single wagon-load if the coke contained more than 2 per cent. of moisture, and fortunately this is not often necessary, for a single wagon-load usually represents a consignment for a foundry (when the moisture-content rarely exceeds 2 per cent.) or for domestic use (when the moisture content, though important, is not often a matter of dispute). When the taking of the number of increments necessary to ensure the standard degree of accuracy (99 out of 100 tests to be within 1 per cent. of the true value) presents difficulty owing to the smallness of the consignment, the number can be halved. The moisture-content as determined on the gross sample should then be within 1·4 per cent. of the true value in 99 tests out of 100, and within 0·37 per cent. in 50 out of 100.

The fact must be emphasized that accuracy in sampling for the moisture-content of consignments of coke delivered over a period, to avoid dispute between the buyer at the blast-furnaces and the seller at the coke-ovens, depends essentially on taking a large number of increments to form the gross sample. For coke from a particular plant, made and quenched in a routine manner, the "average deviation" will not vary much, and it is satisfactory to spread the increments over a period provided that the average moisture-content is based on not less than the total number of increments specified for the particular quality of coke. For example, assuming daily deliveries over one week of a coke containing on the average 5 per cent. of moisture, 95 increments, each not less than 2½ lb. in weight, would be required to form the gross sample for the "standard" accuracy. These increments could be spread over 6 days, so that 16 would be taken daily. Since the moisture-content of the coke would alter during storage, determinations should be made daily (on the 16 increments treated as a gross sample) and averaged at the end of the week.

For the sale of coke to specification, the average analysis over a period, be it weekly or monthly, is required. The buyer, however, might wish to have the right to reject an occasional wagon-load of abnormally high moisture-content, say, 10 per cent., such as might result from excessive spot-quenching on the coke-bench. An abnormal wagon-load should be detected by its appearance or by an abnormal moisture-content in a daily sample, and special tests should be made.

Reduction of the Gross Sample for Analysis.—Considerable error is due to loss of moisture if the coke is crushed too finely

before analysis. It has been found, also, that the use of a riffle to reduce the size of the gross sample causes loss of moisture, owing to free exposure of the coke to the air as it falls in a stream from the openings of the riffle.

Loss of moisture from coke due to fine crushing is exemplified by the following figures :

Size of Coke.	*Moisture-Content, %.*
Uncrushed ..	4·1
Crushed through ½-in.	4·2
Crushed through ¼-in.	3·1
Crushed through ⅛-in.	2·8

A series of tests with a number of cokes ranging in moisture-content from 3 to 13 per cent. showed that they could be crushed to pass through ½-in. without appreciable loss of moisture. The use of such coarse material for analysis for moisture-content demands modification of the usual method, but it is important to adopt this procedure because error can frequently be traced to the use of finely-crushed coke for the determination.

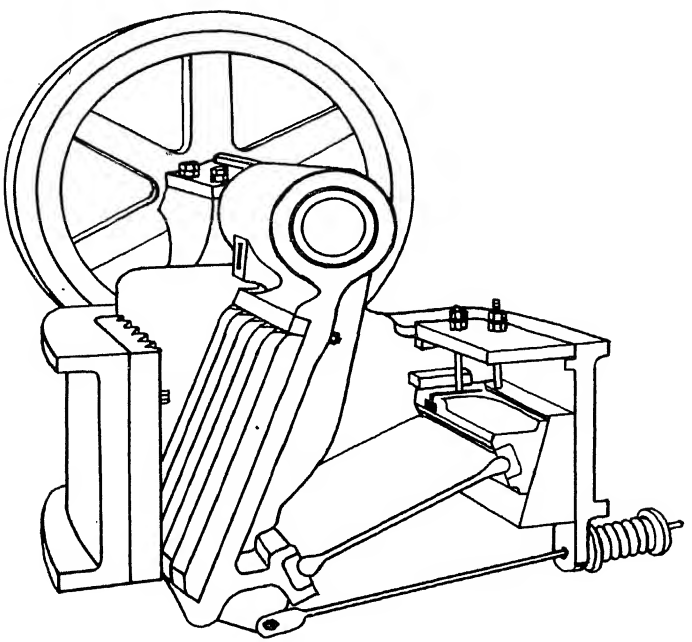


FIG. 11.—Diagrammatic Representation of Jaw Granulator for Crushing Coke to ½-in. Size.

The reduction of the gross sample to through ½-in. size can be done by hand if the practice is followed of dealing separately with the increments taken daily ; but a 250-lb. gross sample, for example, would take about 6 hours to crush by hand and loss of moisture would occur by air drying. It is probably impracticable to deal by hand with a sample weighing more than about 50 lb.

A suitable mechanical crusher has been made for us by Messrs. Hadfields Ltd., as a modification of their standard single toggle granulator. Instead of manganese- or other cast-steel, cast-iron was used, and the opening of the jaw was increased to 10 in. by 5 in. to take as large a size of coke as is usually made. The crusher is illustrated in Fig. 11. It is essentially a jaw crusher with the crushing force applied to the upper end of the movable jaw, which, like the fixed jaw, is serrated. The coke is cut or broken by impact only and no grinding action occurs. With this machine, which is driven by a 4-in. belt at a speed of 250 revolutions per minute by a 5 H.P. motor, 1 cwt. of coke can be crushed to pass 1-in. or ½-in. in 5 minutes. The size of coke produced at different settings of the jaw is as follows :

Setting	Per cent. of Coke passing :					
	1½-in.	1-in.	⅝-in.	½-in.	¼-in.	
Maximum .	90	65	—	17	-	
Coarse .	—	85	55	20	10	6
Fine . .	—	—	95	55	25	15
Minimum .	—	—	—	95	65	40

At the minimum setting the throughput is somewhat restricted, but for all other settings the rate of crushing is the same. It is recommended that a gross sample of coke be crushed to pass ½-in., mixed and reduced to 50 lb. by taking 50 increments of 1 lb. This sample is then mixed and reduced to 2 lb. by taking 20 increments.

Sampling ½-in. Coke for the Determination of Moisture.—Two factors are important when sampling, say, 50 lb. of coke of through ½-in. size to yield the small quantity necessary for a moisture-determination, namely : (*a*) The number of increments by which the small sample is collected, and (*b*) the weight of the small sample. Tests were made to determine the influence of these factors.

(a) The Effect of the Number of Increments.—The weight of the small sample to be obtained was assumed to be 2 lb. A number of 20 lb. samples of different cokes in 2-in and 3-in. pieces were dried, weighed and sprayed with water. After being allowed to drain, the samples were re-weighed (the amount of water they

had absorbed being thus measured) and crushed to through ½-in. size. Samples of 2 lb. were then collected by 5, 10, 15 and 20 increments and the moisture in them determined. The average deviations from the known moisture-contents were :

No. of increments ..	5	10	15	20
Average deviation ..	0·15	0·17	0·14	0·10

A sample taken by 20 increments thus yielded the most accurate result.

(b) The Effect of the Weight of the Sample.—The number of increments by which the samples were to be taken was assumed to be 20. The weights of samples of a through ½-in. coke known to contain 4·1 per cent. of moisture were 2, 1, ½, and ¼ lb. These were found to contain 4·3, 4·0, 4·0 and 3·5 per cent. of moisture.

Similar tests were made by the South Metropolitan Gas Co., with cokes of moisture-content varying between 2·7 and 11·1 per cent. The results can be recorded as average deviations from the true values, thus :

Weight of sample, lb. ..	2	1	½	¼
Average deviation ..	0·22	0·23	0·39	0·33

From these tests it is concluded that the small sample of through ½-in. crushed coke taken for the determination of moisture should be not less than 1 lb. in weight and should be collected by at least 20 increments.

Sampling Coke for the Determination of Ash.—At the outset of work carried out for the British Standards Institution it was assumed that for the correct estimation of the ash in coke, having obtained a 50-lb. sample of through ½-in. size, the following series of operations might have to be carried out :—(i) Reduction of the sample to 25 lb. by "quartering;" (ii) crushing to through ¼-in. and reduction to 6 lb.; (iii) crushing to through ⅛-in. and reduction to 1½-lb.; (iv) crushing to through 14 mesh and reduction to 12 oz.; (v) crushing to through 36 mesh and reduction to 4 oz., and (vi) crushing to through 72 B.S. mesh for analysis.

The necessity for taking each of these steps was examined for a series of cokes ranging from 4·5 to 15 per cent. ash-content. In the first place, with a view to omitting stages (iv) and (v), the procedure outlined up to stage three (iii) was followed, yielding 6-lb. samples of through ⅛-in. size and 4 oz. samples were taken from them by a number of small increments. These were crushed to pass through 60-mesh (72 B.S. mesh) and analysed. The remainders of the 6-lb. samples were treated according to stages (iv) to (vi) before analysis. The results are recorded in Table XIII.

TABLE XIII.—THE SAMPLING OF COKE FOR DETERMINATION OF ASH.

Ash-Content of Coke in 60 mesh Sample		Difference between Duplicate Tests of Samples		Difference between Ash-Content of Samples from $\frac{1}{8}$-in. and from 30-mesh
From $\frac{1}{8}$-in.	From 30-mesh.	From $\frac{1}{8}$-in.	From 30-mesh.	
4·36	4·45	0·01	0·02	0·09
5·00	5·07	nil	0·02	0·07
5·27	5·65	0·06	0·11	0·38
5·65	5·92	0·20	0·15	0·27
7·23	7·00	0·16	nil	0·23
6·82	6·98	0·05	0·15	0·16
8·24	8·00	0·02	nil	0·24
7·95	8·16	0·09	0·01	0·21
8·82	8·73	0·06	0·10	0·09
9·22	9·17	0·01	0·06	0·05
10·73	10·71	0·01	0·01	0·02
12·65	12·88	0·03	0·09	0·23
13·76	13·90	0·10	0·02	0·14
13·54	14·16	0·03	0·02	0·62
	Average difference .	0·06		0·20
	Maximum difference	0·20		0·62

It was concluded that the proposed stages (iv) and (v) of the procedure outlined could be safely omitted, effecting a saving in time of $\frac{1}{2}$-hr. The next step was to determine whether stage (ii) was necessary ; and if not, the sample prepared for the determination of moisture could be used for general analysis.

Comparative tests were therefore made by seven members of the Sampling of Coke Panel of the British Standards Institution with (1) a 1 lb. sample of through $\frac{1}{2}$-in. coke as collected for analysis for moisture and (2) a sample dealt with according to stages (ii) and (iii), both samples being subsequently reduced to 4 oz. of through 60 mesh size for analysis. The average differences found by each experimenter between the ash contents of samples (1) and (2) were : 0·23, 0·52, 0·12, 0·42, 0·33, 0·50 and 0·24, the mean being 0·34 per cent. Such a difference was considered to be too great to allow the 1-lb. " moisture " sample to be used for general analysis. The experimental work had, however, suggested a way of reducing the difference. For, in one series of tests in which cokes made from both coarsely and finely-crushed coal were used, it was found that, in general, the greater differences in ash-content were found in the cokes made from the coarse coal. This observation suggested that the 1-lb. sample of the through $\frac{1}{2}$-in. coke (as taken for the moisture determination) was too small for the correct determination of ash and that a larger sample would give more satisfactory results. This was found to be so in tests made by Dr. G. P. Lishman.

We therefore made experiments in which 1, 2, 4 and 6-lb. samples were taken each by 20 increments, from 50 lb. of through $\frac{1}{2}$-in.

cokes of different ash-contents. These samples (Nos. A1, A2, A3, and A4) were crushed so as to pass $\frac{1}{8}$-in., and 4 oz. samples were then taken from them by 10 increments and crushed to pass a 60-mesh sieve. The rest of the $\frac{1}{2}$-in. coke (37 lb.) was crushed to pass $\frac{1}{4}$-in. and quartered to a sample of 6 lb., which was crushed to pass $\frac{1}{8}$-in. A 4-oz. sample was then taken by 10 increments and crushed to 60-mesh (Sample B). A comparison of the ash analyses is given in Table XIV.

TABLE XIV.—THE SAMPLING OF COKE FOR THE DETERMINATION OF ASH.

True Ash-Content (Sample B)	Deviation shown by Sample:			
	A1 (1-lb.)	A2 (2-lb.)	A3 (4-lb.)	A4 (6-lb.)
23·4	0·39	0·81	0·09	0·09
8·4	0·30	0·20	0·06	0·13
8·8	0·08	nil	0·06	0·10
6·0	0·10	0·20	0·05	0·03
10·0	0·10	nil	0·03	0·02
Average deviation	0·19	0·24	0·06	0·07
Average deviation for cokes of 10 per cent. ash or less	0·32	0·10	0·05	0·02

As a result of these tests it was concluded that, normally, if a 2-lb. sample were taken for the determination of moisture it would serve also for general analysis; but that if the coke had been made from coarse coal or had an abnormally high ash-content, a 4-lb. sample would be preferable.

It has been pointed out that for a true average sample of a consignment of coke to be obtained with respect to its moisture-content the weight of each increment taken should approximate to the average weight of the unbroken pieces in the consignment, which is usually from 2 to 3 lb. It might be objected that this procedure would yield a sample of too low ash content, because the smaller sizes of coke contain the most ash. Although tests reported by E. S. Grumell and A. C. Dunningham to the British Standards Institution showed that no appreciable error in determinations of ash-content is introduced by sampling in the manner suggested, it was considered desirable to test the matter further.

Cokes were sampled at a blast-furnace plant and screened into different sizes below 2-in. Each size was then analysed separately for ash. The results are recorded in Table XV.

Despite the concentration of ash in coke of through 1-in. size, therefore, the exclusion of pieces of less than 2-in. size from the sample taken for analysis does not introduce material error.

This conclusion is of particular moment with regard to samples

taken at ovens, where pieces of typical size can readily be selected. At the blast-furnace, it may be necessary to include pieces of smaller size than 2-in. in the sample taken for analysis, for the sake of the true average moisture-content, for considerable breakage of the coke may have occurred during transport from the ovens.

TABLE XV.—THE EFFECT OF SIZE OF COKE ON ASH-CONTENT.

Coke No.	Ash % in Coke of Size:			
	Over 2-in.	2–1½-in.	1½–1-in.	Through 1-i
1	8·4	8·2	8·1	11·5
2	8·8	8·9	8·6	11·0
3	10·8	10·4	10·0	11·0
4	7·4	7·0	6·9	8·1
5	8·7	8·7	8·8	9·5
6	9·4	8·9	9·1	11·0

By calculation, knowing the proportions of the different sizes, the errors introduced by taking only coke over 1½ or 2-in. in size for the sample were estimated as follows:

Coke No.	Over 1½-in. Size Sampled		Over 2-in. Size Sampled	
	% of coke	Error in ash %	% of coke	Error in ash %
1	88·7	0·20	80·9	0·16
2	86·6	0·07	70·1	0·08
3	81·7	0·03	74·1	0·05
4	76·4	0·04	57·8	0·05
5	75·0	0·13	65·8	0·14
6	70·3	0·41	56·8	0·23
	rror	0·15		0·12
	ı error	0·41		0·23

The Influence of the Weather on the Moisture-Content of Coke.—D. R. Wattleworth (*The Gas World*, 1933, *vol. xlviii, Coking Section, June, page* 8) has recorded determinations of the moisture-content of coke sampled at different levels of a wagon at different dates after loading, which suggest that, unless the exposure has been prolonged unduly, rain does not penetrate more than about 3 ft. below the surface level.

The extent to which moisture is retained depends to a certain extent on the character of the coke. From tests we made on a wagon of Yorkshire coke that had been exposed during 16 days in the middle of winter, the rainfall being 2·2 in., it was evident that the average moisture-content had increased by 0·9 per cent. only, whereas if all the rain had been retained the increase would have been 7·3 per cent. The wagon was sampled at the top and at

1-ft. and 3-ft. depths, 48 increments being taken over each level. The average values for the moisture-content, per cent., were: At the top, 3·1; at 1-ft. level, 2·2; at 3-ft. level, 0·95. When originally loaded the coke contained 0·3 per cent. of moisture.

It is important that the influence of rainfall should not be over-estimated. A rainfall of as much as 1 in. during the normal time of transport of coke from the ovens to the blast-furnaces is unusual; but, if such a rainfall were experienced and all the rain were retained by the coke in a 10-ton wagon, the average moisture-content would be increased by only 2·3 per cent. The coke-maker naturally has no control over any increase in the moisture-content of his coke that may occur during transport, whilst the user pays only for the weight loaded to the wagons. Normally, therefore, both maker and user are concerned only with the moisture-content of the coke as made, so that, whilst sampling of coke from the top of a wagon at the coke-ovens is permissible, at the blast-furnace the lower layers only should be sampled, particularly if there has been much rain whilst the wagon has been in transit. It is comparatively easy to sample only the lower layers of a wagon whilst it is being discharged.

Loss of moisture may also occur from coke exposed to the air, at the surface of a wagon, when the air-current during transport may be important. This was studied by exposing cokes of known moisture-content to still air and to an air-current and weighing them at intervals. The losses of moisture at different periods are recorded in Table XVI as mean values of a number of observations. The losses in 15 minutes from two cokes and from coke nuts are also recorded.

TABLE XVI.—LOSS OF MOISTURE FROM COKE IN AIR, PER CENT.

Time from Start of Test (min.)	5	10	15	30	60
In still air	0·11	0·26	0·32	0·42	0·62
In air-current	0·14	0·35	0·50	0·80	1·28
In air-current for 1 to 2-in. nuts	0·29	0·54	0·75	1·38	2·32
Initial Moisture-Content (per cent.)		2	4	6	8
Loss in still air in 15 min.		0·20	0·37	0·30	0·40
Loss in air-current in 15 min.		0·50	0·55	0·45	0·47
Loss in air-current in 15 min. for coke nuts		0·70	0·65	0·85	0·80

These results show that the loss of moisture from coke on exposure to the air for short periods is not influenced so much by the initial moisture-content as by the time of exposure and the size of the coke.

The effect of such air-drying on a wagon of coke transported 80 miles in fine weather during a period of 8 days was shown by

taking samples from the top, middle and bottom of a wagon. The results were :

Top of wagon	.	3·8 per cent.
Middle of wagon	.	7·1
Bottom of wagon	.	7·0
Mean	.	6·0

The mean moisture-content of the coke as loaded at the ovens was 8·2 per cent.

It is apparent that the upper levels of coke in a wagon may be of abnormal moisture-content, and the sampling of coke from the tops of wagons, after it has left a coke producing plant, cannot therefore be recommended. This is recognised in the revision of the methods for sampling coke issued by the British Standards Institution, in which it is recommended that samples from wagons at the consumers works "shall be taken from freshly-exposed faces," which, by agreement between buyer and seller, may be only from the lower halves of wagons.

Sampling for the Shatter Test.—The shatter test is intended to provide a measure of the liability of cokes to break during handling. The sample of coke to be submitted to the test should therefore be taken before much breakage due to handling has occurred.

With cokes made in modern ovens with rapid coking, the amount of over 2-in. size (the size used in shatter testing) is soon reduced to about half the total weight of the coke by such handling as may be given to it at a blast-furnace plant. It is therefore important that the sample for shatter testing at the blast-furnace plant should be taken from wagons before they are emptied. Moreover, the samples should be taken from the tops of the wagons, since the coke near the bottom has suffered some breakage on its fall through, say, 8 ft. Such a sample will enable an exact comparison to be made with coke leaving a coke-oven plant, which should also be sampled from the tops of wagons, or from a conveyor to the wagons.

A number of pieces ("increments" of about 2½ lb.) should be taken from the consignment, preferably not more than six pieces per wagon, the increments being spread as uniformly as possible over the consignment. The spreading of increments is simple when coke is loaded by a belt conveyor.

The Analysis of Coke

The Determination of Moisture.—Lump coke of ½-in. size retains moisture more tenaciously than does coke powder, and a sample weighing 2 lb. cannot readily be spread as a thin layer. On the other hand, there is no necessity, as there is with coal, to use a low temperature for drying the coke.

Preliminary tests showed that the time required to dry a 2-lb.

sample of coke could be reduced from 9 to 3 hours by heating it at a temperature of 200° C. instead of the usual 105° C., and that there was no oxidation. A series of tests with coke of different sizes and with layers of different thicknesses showed, as might be expected, that drying was completed more rapidly the thinner the layer, being about 2½ hours when the layer was 1½ in. thick and 5 hours when its thickness was 3½ in.

A necessary precaution when determining the moisture in coal by drying it is to cool the sample in a desiccator before weighing. The provision of a desiccator large enough to hold the samples of coke used for these determinations would be inconvenient. Tests were therefore made to see if it was necessary, the 2-lb. samples being weighed (*a*) whilst still hot, and (*b*) after allowing them to cool in air. The average difference in 13 such comparative tests was only 0·15 per cent. It can be concluded, therefore, that the coke samples can be weighed without serious error, whilst hot, a matter of considerable convenience.

The simple procedure for estimating moisture in 2-lb. samples of through ½-in. coke evolved from these tests is to heat them at 200° C. during 3 hours in shallow metal trays, 9 in. in diameter, the coke being in a layer of 1 to 1½ in. It has been found that the time required for drying is not materially affected by differences in the moisture-content between 3 and 16 per cent. Since it is more convenient to use metric weights, a sample weighing 1,000 gm. (rather more than 2 lb.) may be taken. Berenger scales, weighing up to 3 kg. by 0·5 gm., are convenient. A serviceable drying-oven is a gas cooker, with the movable solid shelf set near the bottom of the oven, several trays of coke being placed on the barred shelves in the upper section where a suitable temperature is maintained.

The Determination of Ash.—A factor of considerable importance in the correct estimation of the ash-content of a coke is the possible contamination of the sample by the material of the appliances used to reduce it to a suitable degree of fineness. Such contamination is less likely during reduction to through ½-in. size than when the finer sizes are being prepared, but must be avoided throughout the operations. For a preliminary crushing to through ½-in. size, a plate and stamp of manganese steel can be used, but the further reduction is more expeditiously effected with a 6 in. mortar and a pestle of manganese steel, the pestle having a wooden handle 4 ft. long (see Fig. 12). A pounding action should be used, a rubbing action being avoided. A 4-oz. sample of through ½-in. coke can be reduced to 60 mesh size (72 British Standard mesh) in 30 minutes. The extent to which contamination of the sample may occur if unsuitable appliances are used is illustrated by the results in Table XVII (Coke for Blast Furnaces, p. 58). Grinding in an agate mortar and pestle is regarded as the method least likely to cause contamination.

TABLE XVII.—THE EFFECT ON THE ASH-CONTENT OF COKE OF THE METHOD OF PREPARING THE SAMPLE.

Method of Preparing Sample	Cumberland Coke	Yorkshire Coke	S. Wales Coke
Ground in agate mortar . .	9·4	8·8	6·9
Ground on cast-iron bucking board	11·9	13·3	8·1
Ground in cast-iron mill . .	11·5	9·9	7·7
Ground in hand Wedgewood mill .	—	9·3	7·2
Ground in mechanical Wedgewood mill	9·8	9·2	7·5
Ground in manganese-steel mortar	—	9·0	7·3
Stamped in manganese-steel mortar	9·4	8·7	7·0

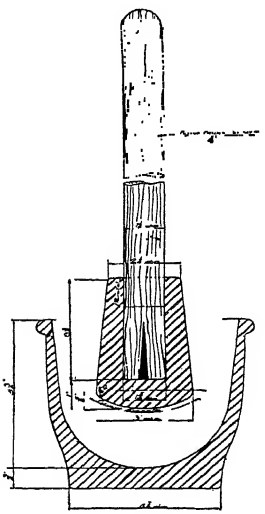


FIG. 12.—Six-inch Manganese-Steel Mortar and Long-handled Pestle for Pulverising Coke.

The actual determination of ash presents but little difficulty. Coke is more difficult to burn than coal, and it is important that the sample taken for analysis should be finely powdered and spread as a thin layer in a shallow dish. One gram is burnt in an oxidising atmosphere (as in a muffle furnace) at a temperature of 750-800° C. until the residue is constant in weight, an operation taking 1 to 1½ hours. Heating should be neither excessive nor prolonged (so that both the temperature and the time must be controlled), or the ash may begin to decompose.

The Determination of Volatile Matter.—The determination of volatile matter in coke requires considerable care. The standard "crucible" method used for coal would give too high a value because the coke would oxidise, insufficient volatile matter being evolved to create a reducing atmosphere. A common method of attempting to prevent this oxidation is to use two crucibles, one within the other, with a layer of coke between the two, but this method is cumbersome.

When the coke is heated it may be oxidised by air either in the crucible or within the muffle. It is therefore advantageous to use a small crucible, of say 15 c.c. capacity, and to displace the air by adding a few drops of benzene. A reducing atmosphere within the muffle can conveniently be created by heating a few lumps of coal in a shallow tray near the entrance, which should have a well-fitting plug-door. As illustrating the necessity for the latter precaution, the following typical volatile matter determinations may be quoted: (*a*) With an oxidising atmosphere in the muffle,

0·83 ; (*b*) with a tray of charcoal at the door of the muffle, 0·73 and (*c*) with a tray of coal at the door of the muffle, 0·70 per cent. For each test a few drops of benzene were added to the crucible.

Allowance must be made for adsorbed moisture in the sample of coke used for the determination of volatile matter. Coke is not so hygroscopic as coal, but when in the form of fine powder dried coke may absorb as much moisture as there is " volatile matter " in it.

The Determination of Nitrogen.—Tests made in the Department of Fuel Technology of Sheffield University (see A. E. Beet, *Fuel in Science and Practice*, 1932, **11**, 304) have shown that the Kjeldahl method commonly used for determining nitrogen in coal is not satisfactory for coke because of the long time required for its digestion with sulphuric acid. A rapid method has been developed as follows :

One gram of the coke, ground to pass a 72 B.S. sieve, is mixed with ½ gram of dry, powdered soda-lime, charged into an unglazed porcelain boat, 90 mm. long and 13 mm. wide, and covered with a layer of ½ gram of soda-lime. The boat is placed in a silica combustion-tube, 24 in. long and ¾ in. in diameter, which is heated at a temperature of 900° C., and steam is passed over it at a rate of 2 grams per minute. The exit end of the combustion tube is well cooled and leads into a cooled flask containing 10 c.c. of 5 per cent. sulphuric acid, where ammonia, produced from the nitrogen in the coke, is absorbed. The ammonia is distilled into standard sulphuric acid and titrated.

The Determination of Carbon.—It is customary at blast furnace plants to calculate the carbon-content of different cokes by subtracting from 100 the sum of the moisture, ash, sulphur and volatile matter. These calculations have the disadvantage that the ash, sulphur and volatile matter are not always recalculated to the " moist " basis. They also ignore the nitrogen and the fact that all the hydrogen as well as the oxygen is not expelled as volatile matter. The true carbon-content may be determined together with the hydrogen-content, by combustion in oxygen, the method being similar to that employed for coal (see *B.S.* 687, " *The Ultimate Analysis of Coal and Coke*"), though a test with coke could be carried out more rapidly than with coal owing to its low hydrogen-content. The same report should be consulted for methods for the determination of phosphorus and arsenic in coke.

APPENDIX

TESTS OF THE MOISTURE-CONTENTS OF COKES QUENCHED IN DIFFERENT WAYS.

A. Quenching Coke by Water-Spray.

TABLE XVIII.—TEST NO. I. VARIATION OF MOISTURE CONTENT OF 48 INCREMENTS OF 1 LB. (TWO PASSES UNDER SPRAY.)

Sample No.	Moisture %	Deviation from Mean	Sample No.	Moisture %	Deviation from Mean
1	3·85	+3·15	25	0·45	−0·25
2	2·5	+1·8	26	0·45	−0·25
3	0·5	−0·2	27	0·3	−0·4
4	0·3	−0·4	28	0·3	−0·4
5	1·4	+0·7	29	0·25	−0·45
6	1·3	+0·6	30	0·5	−0·2
7	0·5	−0·2	31	0·55	−0·15
8	0·6	−0·1	32	0·3	−0·4
9	0·5	−0·2	33	0·45	−0·25
10	0·9	+0·2	34	0·2	−0·5
11	0·3	−0·4	35	0·2	−0·5
12	0·2	−0·5	36	nil	−0·7
13	0·5	−0·2	37	0·2	−0·5
14	0·7	nil	38	0·65	−0·05
15	0·3	−0·4	39	0·4	−0·3
16	0·3	−0·4	40	0·3	−0·4
17	0·45	−0·25	41	0·3	−0·4
18	0·8	+0·1	42	0·55	−0·15
19	0·4	−0·2	43	0·35	−0·35
20	0·4	−0·3	44	0·6	−0·1
21	2·25	+1·55	45	1·9	+1·2
22	1·2	+0·5	46	0·5	−0·2
23	0·7	nil	47	0·5	−0·2
24	0·2	−0·5	48	1·8	+1·1
			Mean	0·7	Av. Dev.
			Max.	3·85	from mean
			Min.	nil	0·45

TABLE XIX.—TEST NO. II. VARIATION OF MOISTURE CONTENT OF 48 INCREMENTS, EACH OF 5 LB., 2½ LB., AND 1 LB.

(THREE PASSES UNDER SPRAY.)

5 lb. Increments			2½ lb. Increments			1 lb. Increments		
Sample No.	Moisture %	Deviation from Mean	Sample No.	Moisture %	Deviation from Mean	Sample No.	Moisture %	Deviation from Mean
1	10·5	+7·5	51	9·5	+5·85	101	13·1	+8·85
2	3·9	+0·9	52	9·55	+5·90	102	11·55	+7·3
3	6·8	+3·8	53	11·0	+7·35	103	4·6	+0·35
4	4·85	+1·85	54	4·2	+0·55	104	12·6	+8·35
5	3·65	+0·65	55	6·25	+2·60	105	1·3	−2·95
6	1·4	−1·6	56	4·9	+1·25	106	4·65	+0·4
7	1·7	−1·3	57	2·65	−1·00	107	3·35	−0·9
8	2·65	−0·35	58	1·0	−2·65	108	0·9	−3·35
9	1·1	−1·9	59	1·6	−1·05	109	1·7	−2·55
10	1·5	−1·5	60	2·5	−1·15	110	0·9	−3·35
11	3·15	+0·15	61	5·2	+1·55	111	7·0	+2·75
12	1·05	−1·95	62	2·75	−0·90	112	4·85	+0·6
13	3·6	+0·6	63	2·35	−1·30	113	1·7	−2·55
14	4·3	+1·3	64	1·3	−2·35	114	4·7	+0·45
15	3·6	+0·6	65	5·7	+2·05	115	4·6	+0·35
16	1·35	−1·65	66	0·8	−2·85	116	2·3	−1·95
17	0·85	−2·15	67	0·45	−3·20	117	0·5	−3·75
18	3·2	+0·2	68	6·25	+2·60	118	7·1	+2·85
19	5·2	+2·2	69	5·05	+1·40	119	8·25	+4·0
20	0·4	−2·6	70	1·8	−1·85	120	1·0	−3·25
21	0·4	−2·6	71	0·25	−3·40	121	4·05	−0·2
22	1·2	−1·8	72	3·1	−0·55	122	5·0	+0·75
23	1·95	−1·05	73	3·65	nil	123	5·7	+1·45
24	0·2	−2·8	74	1·0	−2·65	124	1·55	−2·7
25	1·0	−2·0	75	1·3	−2·35	125	0·4	−3·05
26	3·05	+0·05	76	2·5	−1·15	126	0·85	−3·4
27	1·55	−1·45	77	2·05	−1·60	127	7·1	+3·85
28	2·4	−0·6	78	0·25	−3·40	128	0·4	−3·85
29	1·9	−2·1	79	0·65	−3·00	129	1·8	−2·45
30	7·2	+4·2	80	6·35	−2·70	130	10·6	+6·35
31	6·2	+3·2	81	4·4	+0·75	131	4·3	+0·05
32	4·6	+1·6	82	3·65	nil	132	3·65	−0·6
33	4·25	+1·25	83	4·6	+0·95	133	1·75	−2·5
34	3·45	+0·45	84	4·85	+1·20	134	6·1	+1·85
35	1·8	−1·2	85	1·9	−1·75	135	1·2	−3·05
36	0·1	−2·9	86	0·35	−3·30	136	0·5	−3·75
37	3·05	+0·05	87	1·5	−2·15	137	1·75	−2·5
38	6·5	+3·5	88	11·1	+7·45	138	4·75	+0·5
39	3·15	+0·15	89	1·2	−2·45	139	8·0	+3·75
40	1·2	−1·8	90	1·5	−2·15	140	5·15	+0·9
41	3·85	+0·85	91	1·2	−2·45	141	2·15	−2·1
42	5·2	+2·2	92	5·8	+2·15	142	2·9	−1·35
43	1·4	−1·6	93	2·55	−1·10	143	1·1	−3·15
44	1·8	−1·2	94	2·4	−1·25	144	0·35	−3·9
45	0·1	−2·9	95	1·05	−2·60	145	0·6	−3·65
46	0·5	−2·5	96	1·7	−1·95	146	3·75	−0·5
47	5·2	+2·2	97	8·65	+5·00	147	7·2	+2·95
48	6·85	+3·85	98	11·4	+7·75	148	14·8	+10·55
Mean Max. Min.	3·0 10·5 0·1	Av. Dev. from mean 1·8	Mean Max. Min.	3·65 11·4 0·25	Av. Dev. from mean 2·45	Mean Max. Min.	4·25 14·8 0·4	Av. Dev. from mean 2·85

TABLE XX.—TEST NO. III. VARIATION OF MOISTURE CONTENT OF INCREMENTS, EACH OF 5 LB., 2½ LB., AND 1 LB.

(FOUR PASSES UNDER SPRAY: 2 FAST, 2 SLOW.)

5 lb. Increments			2½ lb. Increments			1 lb. Increments		
Sample No.	Moisture %	Deviation from Mean	Sample No.	Moisture %	Deviation from Mean	Sample No.	Moisture %	Deviation from Mean
1	15·8	+9·65	51	11·7	+4·9	101	12·2	+4·2
2	11·9	+5·75	52	9·25	+2·45	102	8·5	+0·5
3	8·1	+1·95	53	7·4	+0·6	103	22·5	+14·5
4	4·6	−1·55	54	3·35	−3·45	104	3·75	−4·25
5	8·6	+2·45	55	10·6	+3·8	105	12·0	+4·0
6	12·0	+5·85	56	12·0	+5·2	106	12·0	+4·0
7	2·9	−3·25	57	7·5	+0·7	107	3·1	−4·9
8	2·1	−4·05	58	1·65	−5·15	108	1·5	−6·5
9	9·5	+3·35	59	11·5	+4·6	109	20·1	+12·1
10	6·75	+0·6	60	6·9	+0·1	110	11·9	+3·9
11	12·8	+6·65	61	7·75	+0·95	111	8·8	+0·8
12	5·1	−1·05	62	10·7	+3·9	112	5·5	−2·5
13	14·5	+8·35	63	12·7	+5·9	113	14·0	+6·0
14	6·9	+0·75	64	11·75	+4·95	114	6·0	−2·0
15	3·8	−2·35	65	2·5	−4·3	115	6·0	−2·0
16	1·3	−4·85	66	1·0	−5·8	116	1·2	−6·8
17	8·1	+1·95	67	10·5	−3·7	117	10·7	+2·7
18	8·8	+2·65	68	8·8	+2·0	118	6·35	−1·65
19	3·6	−2·55	69	11·75	+4·95	119	20·0	+12·0
20	0·95	−5·2	70	10·5	+3·7	120	5·8	−2·2
21	1·75	−4·4	71	2·6	−4·2	121	5·8	−2·2
22	6·7	+0·55	72	7·05	+0·25	122	5·25	−2·75
23	3·55	−2·6	73	4·15	−2·65	123	1·7	−6·3
24	2·55	−3·6	74	4·2	−2·6	124	1·15	−6·85
25	3·05	−3·1	75	2·55	−4·25	125	2·25	−5·75
26	7·0	+0·85	76	5·6	−1·2	126	7·2	−0·8
27	3·6	−2·55	77	4·35	−1·45	127	4·35	−3·65
28	10·5	+4·35	78	1·8	−5·0	128	1·9	−6·1
29	9·4	+3·25	79	15·8	+9·0	129	11·9	+3·9
30	2·5	−3·65	80	6·9	+0·1	130	15·2	+7·2
31	1·2	−4·95	81	3·4	−3·4	131	5·2	−2·8
32	3·7	−2·45	82	2·0	−4·8	132	0·85	−7·15
33	7·7	+1·55	83	7·6	+0·8	133	8·1	−0·1
34	6·7	+0·55	84	7·25	+0·45	134	9·9	+1·9
35	1·45	−4·7	85	9·6	+2·8	135	10·6	+2·6
36	8·0	+1·85	86	5·2	−1·6	136	14·8	+6·8
37	7·5	+1·35	87	9·8	+3·0	137	10·3	+2·3
38	11·9	+5·75	88	9·1	+2·3	138	11·3	+3·3
39	2·95	−3·2	89	6·0	−0·8	139	7·95	−0·05
40	7·4	+1·25	90	5·0	−2·2	140	13·3	+5·3
41	6·9	+0·75	91	5·0	−1·8	141	9·3	+1·3
42	2·7	−3·45	92	5·2	−1·6	142	12·7	+4·7
43	0·55	−5·6	93	6·1	−0·7	143	3·8	−4·2
44	8·3	+2·15	94	nil	−6·8	144	0·25	−7·75
45	7·65	+1·5	95	8·5	+1·7	145	5·0	−3·0
46	4·4	−1·75	96	5·0	−1·8	146	5·3	−2·7
47	2·0	−4·15	97	4·2	−2·6	147	3·15	−4·85
48	3·2	−2·95	98	1·65	−5·15	148	3·95	−4·05
Mean	6·15	Av. Dev.	Mean	6·8	Av. Dev.	Mean	8·0	Av. Dev.
Max.	15·8	from mean	Max.	15·8	from mean	Max.	22·5	from mean
Min.	0·55	3·3	Min.	nil	3·05	Min.	0·25	4·3

TABLE XXI.—TEST NO. IV. VARIATION OF MOISTURE CONTENT IN 48 INCREMENTS, EACH OF 5 LB., 2½ LB., AND 1 LB.

(FOUR PASSES UNDER SPRAY.)

5 lb. Increments			2½ lb. Increments			1 lb. Increments		
Sample No.	Moisture %	Deviation from Mean	Sample No.	Moisture %	Deviation from Mean	Sample No.	Moisture %	Deviation from Mean
1	1·6	−7·9	51	10·8	+0·5	101	5·7	−4·9
2	2·8	−6·7	52	7·5	−2·8	102	2·8	−7·8
3	15·4	+5·9	53	7·6	−2·7	103	1·2	−9·4
4	2·4	−7·1	54	6·75	−3·55	104	2·75	−7·85
5	8·6	−0·9	55	11·0	+0·7	105	12·8	+2·2
6	8·65	−0·85	56	15·6	+5·3	106	22·8	+12·2
7	7·0	−2·5	57	4·15	−6·15	107	3·2	−7·4
8	9·05	−0·45	58	14·9	+4·6	108	8·75	−1·85
9	7·05	−2·45	59	8·4	−1·9	109	10·5	−0·1
10	7·6	−1·9	60	9·4	−0·9	110	10·3	−0·3
11	6·8	−2·7	61	4·1	−6·2	111	6·5	−4·1
12	9·2	−0·3	62	8·7	−1·6	112	10·0	−0·6
13	12·6	+3·1	63	11·4	+1·1	113	7·5	−3·1
14	10·25	+0·75	64	9·2	−1·1	114	7·0	−3·6
15	11·2	+1·7	65	7·55	−2·75	115	11·4	+0·8
16	12·55	+3·05	66	10·1	−0·2	116	14·15	+3·55
17	6·35	−3·15	67	7·7	−2·6	117	6·1	−4·5
18	8·2	−1·3	68	10·6	+0·3	118	4·35	−6·15
19	6·8	−2·7	69	3·25	−7·05	119	8·2	−2·4
20	6·9	−2·6	70	8·45	−1·85	120	10·85	+0·25
21	12·5	+3·0	71	10·85	+0·55	121	15·2	+4·6
22	10·2	+0·7	72	13·2	+2·9	122	10·45	−0·15
23	8·0	−1·5	73	9·85	−0·45	123	8·95	−1·65
24	7·05	−2·45	74	4·55	−5·75	124	1·75	−8·85
25	6·0	−3·5	75	11·85	+1·55	125	10·65	+0·05
26	8·0	−1·5	76	6·6	−2·7	126	9·75	−0·85
27	9·15	−0·35	77	8·95	−1·35	127	15·8	+5·2
28	7·2	−2·3	78	6·9	−3·4	128	6·6	−4·0
29	15·3	+5·8	79	12·45	+2·15	129	17·5	+6·9
30	16·8	+7·3	80	15·0	+4·7	130	13·95	+3·35
31	14·8	+5·3	81	10·3	nil	131	12·4	+1·8
32	11·95	+2·4	82	16·1	+5·8	132	19·75	+9·15
33	10·0	+0·5	83	9·95	−0·35	133	15·7	+5·1
34	17·0	+7·5	84	17·7	+7·4	134	9·9	−0·7
35	12·0	+2·5	85	3·7	−6·6	135	12·0	+1·4
36	9·7	+0·2	86	10·4	+0·1	136	4·25	−6·35
37	10·9	+1·4	87	22·5	+12·2	137	6·95	−3·65
38	8·5	−1·0	88	17·5	+7·2	138	4·0	−6·6
39	4·55	−4·95	89	2·4	−7·9	139	16·3	+5·7
40	11·9	+2·4	90	8·1	−2·2	140	17·05	+6·45
41	15·75	+6·25	91	7·6	−2·7	141	12·7	+2·1
42	14·9	+5·4	92	18·9	+8·6	142	24·7	+14·1
43	9·3	−0·2	93	14·85	+4·55	143	17·4	+6·8
44	2·3	−7·2	94	6·75	−3·55	144	2·3	−8·3
45	5·6	−3·9	95	14·3	+4·0	145	10·8	+0·2
46	5·5	−4·0	96	2·65	−7·65	146	10·5	−0·1
47	14·5	+5·0	97	19·9	+9·6	147	17·5	+6·9
48	15·85	+5·85	98	15·1	+4·8	148	17·3	+6·7
Mean	9·5	Av. Dev.	Mean	10·3	Av. Dev.	Mean	10·6	Av. Dev.
Max.	17·0	from mean	Max.	22·5	from mean	Max.	24·7	from mean
Min.	1·6	3·15	Min.	2·4	3·65	Min.	1·2	4·4

TABLE XXII.—TEST NO. V. VARIATION OF MOISTURE CONTENT OF INCREMENTS, EACH OF 5 LB., 2½ LB., AND 1 LB.
(SIX PASSES UNDER SPRAY: 4 FAST, 2 SLOW.)

5 lb. Increments			2½ lb. Increments			1 lb. Increments		
Sample No.	Moisture %	Deviation from Mean	Sample No.	Moisture %	Deviation from Mean	Sample No.	Moisture	Deviation from Mean
	17·5	+3·45	51	23·5	+7·8	101	17·4	+2·05
	16·4	+2·35	52	23·3	+7·6	102	6·05	−9·30
	14·2	+0·15	53	16·65	+0·95	103	20·7	+5·35
	14·1	+0·05	54	17·3	+1·6	104	13·35	−2·0
	16·2	+2·15	55	17·2	+1·5	105	20·7	+5·35
	17·7	+5·65	56	18·85	+3·15	106	20·2	+4·85
	17·9	+5·85	57	15·8	+0·1	107	17·0	+1·65
	10·9	−3·15	58	11·7	−4·0	108	22·1	+6·75
9	12·2	−1·85	59	13·95	−1·75	109	15·1	−0·25
10	20·9	+6·85	60	22·8	+7·1	110	16·15	+0·8
11	15·65	+1·6	61	20·0	+4·3	111	22·6	+7·25
12	9·8	−4·25	62	10·6	−4·1	112	19·9	+4·55
13	14·7	+0·65	63	14·0	−1·7	113	22·6	+7·25
14	15·4	+1·35	64	20·5	+4·8	114	20·1	+4·75
15	14·6	+0·55	65	15·6	−0·1	115	18·5	+3·15
16	18·5	+4·55	66	17·6	+1·9	116	15·9	+0·55
17	18·6	+4·55	67	17·1	+1·4	117	16·9	+1·55
18	14·0	−0·05	68	18·45	+2·75	118	17·8	+2·45
19	16·05	+2·0	69	16·25	+0·55	119	21·4	+6·05
20	9·8	−4·25	70	13·1	−2·6	120	9·7	−5·65
21	17·6	+3·55	71	18·5	+2·8	121	19·55	+4·2
22	13·7	−0·35	72	21·3	+5·6	122	14·5	−0·85
23	14·55	+0·5	73	17·1	+1·4	123	19·1	+3·75
24	13·3	−0·75	74	16·3	+0·6	124	17·8	+2·45
25	15·7	+1·65	75	15·7	+0·0	125	14·0	−1·35
26	10·3	−3·75	76	14·5	−1·2	126	7·1	−8·25
27	11·5	−2·55	77	15·4	−0·3	127	12·8	−2·55
28	11·0	−3·03	78	10·9	−4·8	128	11·3	−4·05
29	25·8	+11·75	79	20·75	+5·05	129	16·0	+0·65
30	16·2	+2·15	80	9·8	−5·9	130	18·1	+2·75
31	12·6	−1·45	81	17·7	+2·0	131	16·55	+1·2
32	9·95	−4·1	82	13·4	−2·3	132	9·6	−5·75
33	16·85	+2·8	83	19·0	+3·3	133	21·9	+6·55
34	17·9	+3·85	84	16·5	+0·8	134	18·1	+2·75
35	13·1	−0·95	85	6·6	−9·1	135	4·55	−10·8
36	4·75	−9·3	86	13·95	−1·75	136	8·7	−6·65
37	17·75	+3·7	87	18·2	+2·5	137	21·5	+6·15
38	25·4	+11·35		17·5	+1·8	138	21·1	+5·75
39	12·5	−1·55		19·0	+3·3	139	19·95	+4·6
40	8·85	−5·2	90	7·15	−8·55	140	6·70	−8·65
41	12·85	−1·2	91	12·9	−2·8	141	7·0	−8·25
42	8·45	−5·6	92	13·75	−1·95	142	9·65	−5·7
43	14·6	+0·55	93	10·4	−5·3	143	8·1	−7·25
44	9·75	−4·3	94	7·1	−8·6	144	8·8	−6·55
45	12·6	−1·45	95	19·35	+3·65	145	20·9	+5·55
46	11·0	−3·05	96	22·35	+6·65	146	22·8	+7·45
47	4·95	−9·1	97	10·7	−5·0	147	11·95	−3·45
48	3·60	−10·45	98	4·7	−11·0	148	5·40	−9·95
Mean	14·05	Av. Dev.	Mean	15·7	Av. Dev.	Mean	15·35	Av. Dev.
Max.	25·8	from mean	Max.	23·5	from mean	Max.	22·8	from mean
Min.	3·6	3·45	Min.	4·7	3·5	Min.	4·55	4·7

B. Quenching Coke Centrally.

TABLE XXIII.—VARIATION OF MOISTURE CONTENT OF 48 INCREMENTS, EACH OF 5 LB., 2½ LB., AND 1 LB.

(4,000 GALLONS OF WATER DISCHARGED IN 35 SEC. ON TO 8 TONS OF COKE.)

5 lb. Increments			2½ lb. Increments			1 lb. Increments		
Sample No.	Moisture %	Deviation from Mean	Sample No.	Moisture %	Deviation from Mean	Sample No.	Moisture %	Deviation from Mean
1	0·5	−3·1	51	1·95	−1·5	101	8·2	+3·35
2	3·8	+0·2	52	nil	−3·45	102	3·85	−1·0
3	1·8	−1·8	53	5·9	+2·45	103	0·6	−4·25
4	1·8	−1·8	54	0·6	−2·85	104	1·55	−3·3
5	5·6	+2·0	55	2·8	−0·65	105	5·25	+0·4
6	0·65	−2·95	56	7·2	+3·75	106	0·85	−4·0
7	2·4	−1·2	57	2·4	−1·05	107	9·6	+4·75
8	3·0	−0·6	58	5·05	+1·6	108	9·35	+4·5
9	10·4	+6·8	59	0·65	−2·8	109	2·2	−2·65
10	3·2	−0·4	60	2·2	−1·25	110	4·55	−0·3
11	6·4	+2·8	61	0·55	−2·9	111	2·3	−2·55
12	3·0	−0·6	62	1·5	−1·95	112	1·3	−3·55
13	5·7	+2·1	63	2·75	−0·7	113	3·05	−1·2
14	3·2	−0·4	64	2·9	−0·55	114	5·75	+0·9
15	0·6	−3·0	65	1·25	−2·2	115	1·2	−3·65
16	7·8	+4·2	66	1·0	−2·45	116	3·2	−1·05
17	2·7	−0·9	67	0·9	−2·55	117	5·65	+0·8
18	0·4	−3·2	68	2·0	−1·45	118	12·8	+7·95
19	2·7	−0·9	69	6·3	+2·85	119	5·0	+0·15
20	8·2	+4·6	70	2·65	−0·8	120	2·5	−2·35
21	6·05	+2·45	71	5·35	+1·9	121	6·7	+1·85
22	3·2	−0·4	72	0·75	−2·7	122	2·0	−2·85
23	0·95	−2·65	73	3·5	+0·05	123	3·9	−0·95
24	5·7	+2·1	74	3·15	−0·3	124	1·25	−3·6
25	0·9	−2·7	75	0·1	−3·35	125	2·4	−2·45
26	4·15	+0·55	76	2·8	−0·65	126	3·45	−1·4
27	3·95	+0·35	77	7·25	+3·8	127	6·75	+1·9
28	11·15	+7·55	78	0·2	−3·25	128	5·8	+0·95
29	4·9	+1·3	79	6·6	+3·15	129	10·9	+6·05
30	4·1	+0·5	80	2·2	−1·25	130	4·5	−0·35
31	2·2	−1·4	81	2·7	−0·75	131	6·05	+1·2
32	2·55	−1·05	82	4·1	+0·65	132	3·35	−1·5
33	5·0	+1·4	83	8·0	+4·55	133	10·35	+5·5
34	0·6	−3·0	84	7·8	+4·35	134	11·9	+7·05
35	5·85	+2·25	85	8·75	+5·3	135	3·3	−1·55
36	7·5	+3·9	86	4·05	+0·6	136	12·1	+7·25
37	2·3	−1·3	87	2·6	−0·85	137	0·6	−4·25
38	7·45	+3·85	88	6·2	+2·75	138	2·25	−2·6
39	2·6	−1·0	89	6·8	+3·35	139	1·7	−3·15
40	1·3	−2·3	90	4·5	+1·05	140	1·75	−3·1
41	1·1	−2·5	91	7·95	+4·5	141	13·5	−3·5
42	2·2	−1·4	92	0·85	−2·6	142	2·95	−1·9
43	1·85	−1·75	93	1·4	−2·05	143	1·1	−3·75
44	3·55	−0·05	94	3·15	−0·3	144	3·65	−1·2
45	1·9	−1·7	95	3·55	+0·1	145	6·5	+1·65
46	3·6	nil	96	7 05	+3·6	146	9·3	+4·45
47	0·7	−2·9	97	2·7	−0·75	147	14·4	+9·55
48	1·8	−1·8	98	0·7	−2·75	148	3·6	−1·25
Mean Max. Min.	3·6 11·15 0·4	Av. Dev. from mean 2·05	Mean Max. Min.	3·45 8·75 nil	Av. Dev. from mean 2·1	Mean Max. Min.	4·85 14·4 0·6	Av. Dev. from mean 2·9

C. Quenching Coke by Hand Hose.

TABLE XXIV.—VARIATION OF MOISTURE CONTENT OF 48 INCREMENTS, EACH OF 5 LB., 2½ LB. AND 1 LB.

(QUENCHED WITH HAND HOSE ON INCLINED BENCH.)

5 lb. Increments			2½ lb. Increments			1 lb. Increments		
Sample No.	Moisture %	Deviation from Mean	Sample No.	Moisture %	Deviation from Mean	Sample No.	Moisture %	Deviation from Mean
1	3·9	−4·7	51	4·8	−4·8	101	3·0	−7·9
2	8·55	−0·05	52	13·85	+4·25	102	16·9	+6·0
3	14·85	+6·25	53	4·7	−4·9	103	21·3	+10·4
4	4·35	−4·25	54	8·4	−1·2	104	10·7	−0·2
5	4·25	−4·35	55	12·1	+2·5	105	9·3	−1·6
6	11·25	+2·65	56	10·4	+0·8	106	12·7	+1·8
7	2·1	−6·5	57	10·9	+1·3	107	18·7	+7·8
8	6·4	−2·2	58	5·85	−3·75	108	2·8	−8·1
9	15·25	+6·65	59	8·7	−0·2	109	19·4	+8·5
10	5·0	−3·6	60	14·7	+5·1	110	0·5	−10·4
11	3·2	−5·4	61	—	—	111	3·9	−7·0
12	13·95	+5·35	62	12·9	+3·3	112	12·2	+1·3
13	10·0	+1·4	63	5·5	−4·1	113	14·4	+3·5
14	4·3	−4·3	64	16·7	+7·1	114	10·9	+9·0
15	5·2	−3·4	65	1·6	−8·0	115	4·1	−6·8
16	2·2	−6·4	66	18·5	+8·9	116	5·2	−5·7
17	7·9	−0·7	67	11·3	+1·7	117	19·1	+8·2
18	9·65	+1·05	68	14·2	+4·6	118	19·6	+8·7
19	17·5	+8·9	69	11·4	+1·8	119	7·8	−3·1
20	5·7	−2·9	70	2·75	−6·85	120	6·9	−4·0
21	7·2	−1·4	71	5·8	−3·8	121	2·1	−8·8
22	19·2	+10·6	72	5·7	−3·9	122	16·0	+5·1
23	8·9	+0·3	73	17·5	+7·9	123	32·1	+21·2
24	5·8	−2·8	74	5·45	−4·15	124	8·3	−2·6
25	4·65	−3·95	75	8·25	−1·35	125	18·4	+7·5
26	7·9	−0·7	76	2·85	−6·75	126	11·2	+0·3
27	6·3	−2·3	77	3·65	−5·95	127	7·6	−3·3
28	9·15	+0·55	78	10·7	+1·1	128	10·2	−0·7
29	5·85	−2·75	79	3·05	−6·55	129	0·8	−10·1
30	6·9	−1·7	80	17·8	+8·2	130	19·8	+8·9
31	18·15	+9·55	81	8·7	−0·2	131	10·7	−0·2
32	4·05	−4·55	82	7·7	−1·2	132	1·3	−9·6
33	2·75	−5·85	83	8·75	−0·85	133	2·8	−8·1
34	3·95	−4·65	84	19·0	+9·4	134	18·2	+7·3
35	13·5	+4·9	85	8·8	−0·8	135	18·0	+7·1
36	9·25	+0·65	86	4·8	−4·8	136	7·1	−3·8
37	10·2	+1·6	87	17·4	+7·8	137	22·0	+11·1
38	9·55	−0·85	88	9·5	−0·1	138	18·5	+7·6
39	9·7	+1·1	89	12·2	−2·6	139	25·0	+14·1
40	5·75	−2·85	90	7·2	−2·4	140	9·0	−1·9
41	5·1	−3·5	91	21·3	+11·7	141	6·5	−4·4
42	14·8	+6·2	92	5·65	−3·95	142	8·0	−2·9
43	7·45	−1·15	93	14·6	+5·0	143	4·3	−6·6
44	11·55	+2·95	94	8·0	−1·6	144	0·7	−10·2
45	14·8	−6·2	95	7·75	−1·85	145	9·9	−1·0
46	14·15	+5·55	96	15·0	+5·4	146	3·6	−7·3
47	6·95	−1·65	97	2·85	−6·75	147	2·9	−8·0
48	14·45	+5·85	98	11·2	+1·6	148	1·7	−9·2
Mean	8·6	Av. Dev.	Mean	9·6	Av. Dev.	Mean	10·9	Av. Dev.
Max.	19·2	from mean	Max.	21·3	from mean	Max.	32·1	from mean
Min.	2·1	3·5	Min.	1.6	4·05	Min.	0·5	6·4

TABLE XXV.—VARIATION OF MOISTURE CONTENT OF 48 INCREMENTS, EACH OF 5 LB., 2½ LB. AND 1 LB.

(SECOND TEST AT PLANT USING QUENCHING BY HAND HOSE ON AN INCLINED BENCH.)

5 lb. Increments			2½ lb. Increments			1 lb. Increments		
Sample No.	Moisture %	Deviation from Mean	Sample No.	Moisture %	Deviation from Mean	Sample No.	Moisture %	Deviation from Mean
1	16·35	+6·25	51	12·8	+1·65	101	4·3	−4·2
2	10·5	+0·4	52	18·1	+6·95	102	5·25	−3·25
3	21·8	+11·7	53	22·2	+11·5	103	4·05	−4·45
4	6·0	−4·1	54	1·95	−9·2	104	23·0	+14·5
5	5·7	−4·4	55	6·4	−4·75	105	6·5	−2·0
6	6·7	−3·4	56	12·7	+1·55	106	10·6	+2·1
7	14·2	+4·1	57	14·7	+3·55	107	7·8	−0·7
8	7·85	−2·25	58	13·9	+2·75	108	30·6	+22·1
9	5·2	−4·9	59	8·5	−2·65	109	5·5	−3·0
10	3·8	−6·3	60	6·4	−4·75	110	7·5	−1·0
11	2·65	−7·45	61	11·1	−0·05	111	4·55	−3·95
12	7·55	−2·55	62	7·45	−3·7	112	0·4	−8·1
13	8·5	−1·6	63	11·2	+0·05	113	6·1	−2·4
14	8·2	−1·9	64	15·9	+4·75	114	8·65	+0·15
15	15·0	−4·2	65	28·1	+16·95	115	1·6	−6·9
16	3·3	−6·8	66	11·9	+0·75	116	18·5	+10·0
17	3·7	−6·4	67	3·25	−7·9	117	6·15	−2·35
18	13·05	+2·95	68	21·2	+10·05	118	12·2	+3·7
19	13·0	+2·9	69	12·0	+0·85	119	0·5	−8·0
20	3·75	−6·35	70	5·65	−5·5	120	0·7	−7·8
21	2·85	−7·25	71	18·65	+7·5	121	17·6	+9·1
22	17·65	+7·55	72	15·05	+3·9	122	4·05	−4·45
23	14·95	+4·85	73	10·65	−0·5	123	4·8	−3·7
24	3·35	−6·75	74	16·85	+5·7	124	28·0	+19·5
25	9·35	−0·75	75	2·0	−9·15	125	9·3	+0·8
26	16·95	+6·85	76	13·35	+2·2	126	0·5	−8·0
27	2·7	−7·4	77	7·15	−4·0	127	5·3	−3·2
28	15·8	+5·7	78	11·0	−0·15	128	11·3	+2·8
29	13·8	+3·7	79	10·45	−0·7	129	9·6	+1·1
30	11·95	+1·85	80	13·55	+2·4	130	1·65	−6·85
31	15·85	+5·75	81	8·2	−2·95	131	4·6	−3·9
32	3·15	−6·95	82	7·75	−3·4	132	5·3	−3·2
33	5·4	−4·7	83	16·85	+5·7	133	11·3	+2·8
34	0·5	−9·6	84	11·85	+0·7	134	17·0	+8·5
35	5·15	−4·95	85	8·65	−2·5	135	2·25	−5·95
36	10·0	−0·1	86	12·85	+1·7	136	3·35	−5·15
37	13·8	+3·7	87	6·5	−5·65	137	3·25	−5·25
38	5·3	−4·8	88	3·35	−7·8	138	2·85	−5·65
39	7·3	−2·8	89	16·75	+5·6	139	3·5	−5·0
40	10·6	+0·5	90	7·25	−3·9	140	12·8	+4·3
41	16·05	+5·95	91	16·1	+4·95	141	13·05	+4·55
42	12·1	+2·0	92	4·85	−6·3	142	21·9	+13·4
43	18·05	+7·95	93	10·55	−0·6	143	0·75	−7·75
44	11·15	+1·05	94	6·45	−4·7	144	8·2	−0·3
45	7·45	−2·65	95	4·9	−6·25	145	0·5	−8·0
46	8·5	−1·6	96	19·85	+8·7	146	16·75	+8·25
47	24·5	+14·4	97	2·85	−8·3	147	17·9	+9·4
48	24·7	+14·6	98	6·0	−5·15	148	7·9	−0·6
Mean	10·1	Av. Dev.	Mean	11·15	Av. Dev.	Mean	8·5	Av. Dev.
Max.	24·7	from mean	Max.	28·2	from mean	Max.	30·6	from mean
Min.	0·5	4·85	Min.	1·9	4·6	Min.	0·4	5·65

CHAPTER VIII

THE DENSITY AND POROSITY OF COKE

METHODS FOR DETERMINING DENSITY AND POROSITY

Apparent Density

Displacement and Boiling Methods.—In "Coke for Blast Furnaces," Part I, Chapter V, two methods for determining the apparent density of coke were described, namely (1) by simple displacement of water (the "displacement method"), and (2) by displacement after preliminary boiling and cooling in water, to expel air from open pores (the "boiling method"). These methods are simple, but the choice of a full-length specimen of coke to represent the average quality of a consignment may be difficult.

Table XXVI.—The Apparent Density of Coke.

Sample No.	Apparent Density	Deviation from Mean.	Sample No.	Apparent Density.	Deviation from Mean.
1	0·84	0·04	26	1·03	0·15
2	0·87	0·01	27	0·82	0·06
3	0·88	0·00	28	0·88	0·00
4	0·90	0·02	29	0·88	0·00
5	0·91	0·03	30	0·83	0·05
6	0·87	0·01	31	0·91	0·03
7	0·85	0·03	32	0·86	0·02
8	0·86	0·02	33	0·89	0·01
9	0·90	0·02	34	0·90	0·02
10	0·87	0·01	35	0·89	0·01
11	0·89	0·01	36	0·86	0·03
12	0·92	0·04	37	0·89	0·01
13	0·89	0·01	38	0·90	0·02
14	0·85	0·03	39	0·79	0·09
15	0·83	0·05	40	0·83	0·05
16	0·89	0·01	41	0·85	0·03
17	0·88	0·00	42	0·86	0·02
18	0·88	0·00	43	0·86	0·02
19	0·94	0·06	44	0·89	0·01
20	0·90	0·02	45	0·94	0·06
21	0·95	0·07	46	0·90	0·02
22	0·91	0·03	47	0·89	0·01
23	0·86	0·02	48	0·89	0·01
24	0·90	0·02	49	0·86	0·02
25	0·87	0·01	50	0·89	0·01
Mean	0·88	0·023	Mean	0·88	0·030

The variability in the apparent density of a coke was found by making separate determinations on 50 full-length pieces, using the boiling method. The coke was made from Parkgate coal top-charged to a 20-in. oven and carbonised in 28 hours. The results, together with the deviations of individual determinations from the mean value, are recorded in Table XXVI.

The range of the results was 0·79 to 1·03, but 78 per cent. of the determinations lay between 0·85 and 0·91 with a mean value

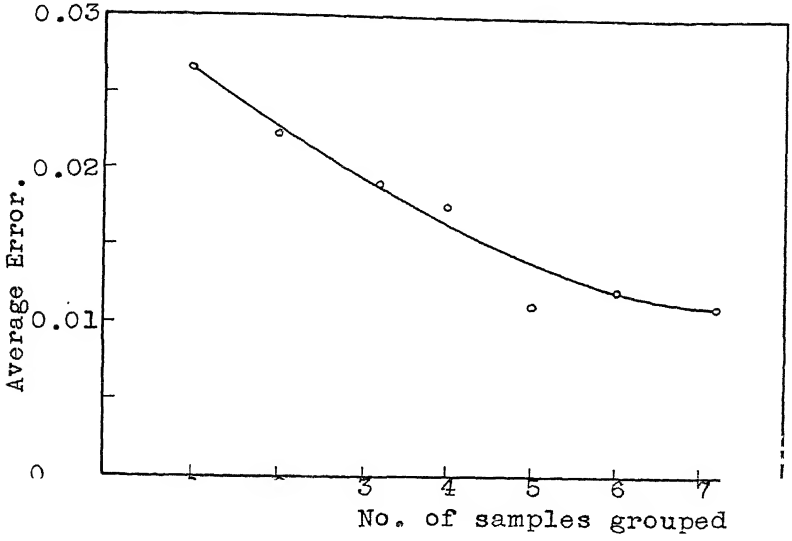


FIG. 13.—Average Error of the Mean Apparent Specific Gravity for Various Groups of Tests.

of 0·88. The average deviation from the mean was 0·027. Fig. 13 shows the average deviation of groups of from 1 to 7 pieces in their order of examination. It will be seen that the value for the average deviation falls from 0·027 to 0·011 as the "unit" for the test is increased from 1 to 7 pieces. From the theory of errors, it can be shown that 77 pieces of this coke (of average weight 2½-lb.) would have to be tested to attain an accuracy, 99 times out of 100, of ±0·01. For an accuracy of 0·02, 19 tests would be required.

A statistical analysis of determinations of apparent density of 200 cokes has been made, half of them with four pieces and half with five, each piece being tested separately. For each coke, the mean value and the deviation of each value from the mean, and hence the average deviation, was calculated. The results are recorded in Table XXVII on a percentage basis.

TABLE XXVII.—APPARENT DENSITIES. PERCENTAGES OF COKES HAVING DIFFERENT AVERAGE DEVIATIONS FROM MEAN VALUES.

No. of Tests Averaged.	Average Deviation from Mean.										
	0·00	0·01	0·02	0·03	0·04	0·05	0·06	0·07	0·08	0·09	0·10
4 ...	2	13	27	17	18	9	6	2	3	2	2
5 ...	2	17	36	17	18	4	3	1	2	0	0

	Mean Avge. Deviation.	Max. Avge. Deviation.	Minimum Avge. Deviation.	Range.
4 ...	0·034	0·10	0·00	0·10
5 ...	0·028	0·08	0·00	0·08

The average deviations are lower when the mean of 5 tests is taken, 90 per cent. being 0·04 or less as compared with 77 per cent. when only 4 tests are made. The typical average deviation, *i.e.* the one for which the highest percentage of results is found, is 0·02 (Table XXVII).

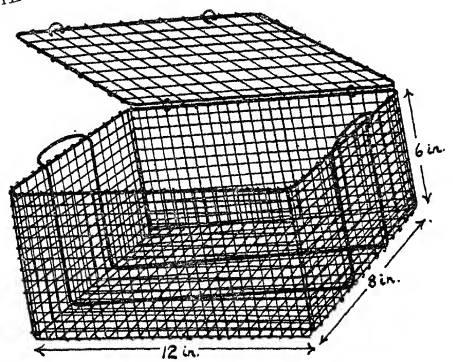
The variability of coke with respect to its apparent density is thus such that many pieces may have to be tested to arrive at an accuracy within 0·02 of the true value. We have usually tested 4 or 5 pieces, and the error, though most often 0·02, may have been 0·04 or greater. For a coke of apparent density 0·88 the results of the examination of 5 pieces might lie within the range 0·84 to 0·92. If the real density of this coke were 1·92 the percentage porosity might then be recorded, according to the value taken for the apparent density, as :

Apparent density	0·84	0·86	0·88	0·90	0·92
Porosity, per cent.	56·3	55·2	54·1	53·1	52·1

There thus might be an error of ± 2 per cent. in the value recorded for the total porosity.

The range of values for the total porosity of high-temperature cokes is narrow, about 50 to 55 per cent. To be significant, the value should not be subject to error of more than 1 per cent. This in turn demands that the error in the determination of apparent density should not exceed 0·01. We have seen that with cokes similar to the Parkgate coke examined (Table XXVI) this means that tests on at least 77 full-length pieces should be made. With more variable cokes a larger number of pieces should be examined. We conclude that when only 4 or 5 pieces of coke are tested by the "boiling method" for apparent density the results of calculations of total porosity are of doubtful value.

It seemed possible that the use of broken coke, instead of full-length pieces, might reduce the average deviation of each test result from the mean, and thus give a sufficiently accurate result from a comparatively small number of tests. What has been termed the "cage method" of test (a modification of the "displacement" and "boiling" methods, using broken coke) was therefore studied.



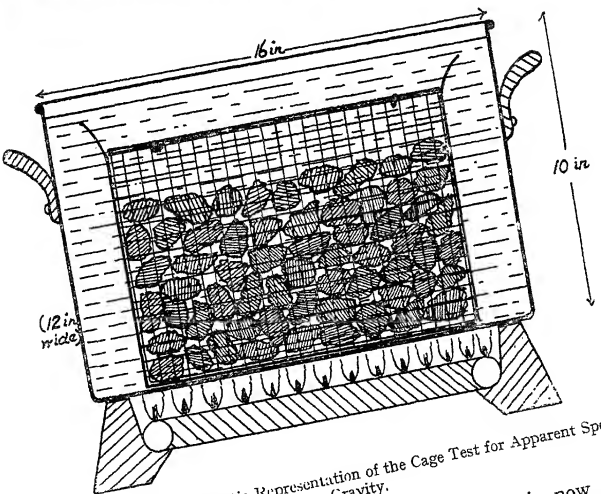


FIG. 14.—Diagrammatic Representation of the Cage Test for Apparent Specific Gravity.

The Cage Method.—Since the shatter test is now widely used, it was considered that coke broken in that test should be taken for determinations of apparent density, so that values for coke of known shatter indices would be recorded. A weight of about 10-lb. of broken coke (equivalent to about 4 full-length pieces) was found convenient to handle in a wire cage designed for the purpose. The cage was of ½-in. mesh iron wire, 12-in. by 8-in. by 6-in. deep, with a lid of the same mesh wired on. (Fig. 14.)

The weighed cage, containing a weighed quantity of about 10 lb. of coke screened over ½-in., from a shatter test, was boiled in water during ½-hour and allowed to cool in water. The weights of the cage full of coke (*a*) in cold water and (*b*) in air (after draining) were then determined and the apparent density of the coke calcylated from the formula:

$$\text{Apparent density} = \frac{W_1 - w_1}{(W_3 - w_3) - (W_2 - w_2)}$$

in which W_1, W_2 and W_3 are the weights of the cage and coke together in air (dry), in water and in air (wet); and w_1, w_2 and w_3 are the corresponding weights of the cage alone.

Whilst the use of 10 lb. of broken coke in one test was more convenient than the testing of several full-length pieces, it was realised that water might be retained by capillary attraction amongst the smaller pieces. Comparative tests were therefore made with the same cokes in which the ½–1 in., 1–1½-in. and 1½–2-in. sizes had been successively screened out. Three cokes were tested in this manner. The results are recorded in Table XXVIII.

TABLE XXVIII.—APPARENT DENSITIES. CAGE" TESTS ON THREE COKES.

Size of Coke.	No. 40 A (Lancs.)	No. 44 B (S. Yorks.)	No. 29 C (S. Yorks.)
Over 2-in.	1·02	0·90	0·89
Over 1½-in.	1·01	0·91	0·91
Over 1-in.	1·01	0·90	0·91
Over ½-in.	1·02	0·90	0·91

TABLE XXIX.—APPARENT DENSITIES OF PARKGATE COKE. THE EFFECT OF SIZE OF THE COKE.

Test No.	Size				
	Over 2-in.	2–1½-in.	1½–1-in.	1–½-in.	Less than ½-in.
1	0·89	0·90	0·91	0·91	—
2	0·88	0·90	0·91		
3	0·88	0·91			
4	0·89				
5	0·88				
6	0·86				
7	0·91				
Average	0·884	0·903	0·91	0·91	—
Per cent. of total coke	65·0	17·5	9·6	5·4	1·8

The results did not appear to be affected by retention of water, but it seemed that there might be differences in the apparent density according to the size of the coke. Further tests were therefore made with the Parkgate coke previously used when in full-length pieces (Table XXVI). The dried coke was subjected to shatter test and screened into over 2-in., 2–1½-in., 1½–1-in. and 1–½-in. sizes. "Cage" tests were then made on each size separately. The results are recorded in Table XXIX.

The smaller sizes of coke were thus found to have the higher apparent densities. The average value from the 7 tests of the over 2-in. size (65 per cent. of the total coke) was 0·884, which agrees closely with the average value (0·882) found for 50 full-length pieces. The average deviation from the mean in the 7 "cage" tests with the over 2-in. size was only 0·01, as compared with 0·03 for the results obtained with 50 full-length pieces. A sufficient number (*e.g.* 50) of "cage" tests with one coke has not yet been made to determine the true average error, nor has the number of tests required to ensure accuracy, 99 times out of 100, within ± 0·01 been determined, but it is evident that broken coke provides a more uniform sample than a single lump.

"Cage" tests made with a Derbyshire coke gave the results recorded in Table XXX.

TABLE XXX.—APPARENT DENSITIES. "CAGE" TESTS ON DERBYSHIRE COKE.

	Size				
	Over 2-in.	2–1½-in.	1½–1-in.	1–½-in.	Less than ½-in.
	0·99 0·95 0·90	0·88 0·86	0·86 0·95	0·98	—
Average	0·95	0·92	0·90	0·98	—
Per cent of total coke	38·0	34·8	19·6	12·8	4·0

These results are irregular because the coke itself was irregular in quality, as was disclosed by normal "boiling" tests with full-length pieces of coke taken from the top, middle and bottom of the

	Mean Apparent Density	Average Deviation (4 tests)
Top	0·90	0·005
Middle	0·91	0·03
Bottom	0·98	0·03

The high value for the apparent density of the coke from

the bottom of the oven is due to the fact that the charge was compressed. When the oven was top-charged the values were:

	Mean Apparent Density	Average Deviation (6 tests)
Top	0·83	0·04
Bottom	0·88	0·03

A comparison of the normal "boiling" method and the "cage" method of determining apparent densities has been made by the Northern Coke Research Committee.* Whilst the results obtained by the "boiling" method showed smaller average deviations than are usual for Midland cokes, the results by the "cage" method were throughout superior in this respect. Table XXXI records this comparison.

TABLE XXXI.—COMPARISON OF APPARENT DENSITIES BY "BOILING" AND "CAGE" METHODS (NORTHERN COKE RESEARCH COMMITTEE).

	Boiling Method		Cage Method	
	Apparent Density	Average Deviation	Apparent Density	Average Deviation
Durham coke .	0·90	0·01	0·90	0·003
Scottish coke .	1·02	0·03	1·03	0·007
Welsh coke .	0·87	0·02	0·85	0·010

Determinations of apparent density made by the South Metropolitan Gas Company for the British Standards Institution with 27 large lumps of horizontal retort coke by the "boiling" method gave a mean value of 0·86 with an average deviation of 0·082. On crushing 1 cwt. of the large coke to pass through a 1-in. screen and removing what passed through ⅜-in., 80 per cent. of 1—⅜-in. size was obtained. Twelve "cage" tests (using about 1 lb. of coke in each test) gave a mean value for the apparent density of 0·889 with an average deviation of 0·009. Unfortunately, the crushing of the large pieces, by the destruction of large pores, caused an increase in the apparent density, amounting to as much as 0·045 in some cases, and 0·017 on the average. Neither the "boiling" nor the "cage" method of determining the apparent density could therefore be regarded as satisfactory for cokes having a spongy structure.

It is thus evident that to obtain a reliable value for the apparent density of coke considerable pains must be taken. The "cage" method of determination is to be preferred because the average

* Private communication.

error is reduced thereby, but even with this method a considerable number of tests (how many has not yet been determined accurately) may have to be made.

A method for determining the apparent density of small pieces of coke is described in Appendix I of this Chapter.

Porosity

The porosity of coke is calculated by the formula :

$$\text{Porosity, per cent.} = 100 \left(\frac{\text{Real S.G.} - \text{App. S.G.}}{\text{Real S.G.}} \right)$$

The porosity of coke is dependent upon (1) the bulk-density of the charge in the oven, (2) the amount of volatile matter driven off from the charge during the plastic stage, and (3) the extent to which shrinkage and impregnation by carbon reduces the size of the pores after plasticity is over.

The effect of the bulk-density of the charge is shown by comparison of top-charges and cake-charges of the same coals :

Coal No.		17A.	18E.	18F.	20B.	21A.	29B.
Porosity, per cent.	Top charge	51·5	54·4	55·6	55·5	51·8	54·2
Porosity, per cent.	Cake charge	45·8	50·3	50·9	50·8	51·0	45·4

Increasing the rate of heating the ovens increases the rate of evolution of volatile matter during plasticity of the charge, and thus increases the porosity of the coke, unless the charge is a blend containing non-coking material, as the results in Table XXXII illustrate :

Table XXXII.—Effect of Rate of Heating on the Porosity of Coke.

Coal No. 18E		Coal No. 29B		Coal No. 29D		Coal No. 29M*	
Rate of Heating, in. per hr.	Porosity, per cent.	Rate of Heating, in. per hr.	Porosity, per cent.	Rate of Heating, in. per hr.	Porosity, per cent.	Rate of Heating, in. per hr.	Porosity per cent
0·63	54·4	0·95	54·2	0·95	52·7	0·86	43·7
1·17	58·9	1·17	55·0	1·08	54·3	1·47	39·0

* A blend of S. Yorkshire coking slack with 30 per cent. of anthracite.

In older coking practice the rate of heating the ovens in different districts was about the same, whereas the method of charging the ovens might be different. Average values for the porosity of cokes from different districts show that for the same method of charging the ovens the porosity was fairly constant. It was always less for cake charges than for top charges, as shown in Table XXXIII :

TABLE XXXIII.—MEAN POROSITY OF COKES FROM TOP AND CAKE CHARGES IN DIFFERENT DISTRICTS.

District	No. of Cokes averaged	Open Pores, per cent.	Total Pores, per cent.
		Top Charges.	
S. Wales .	4	41·8	52·9
Durham .	5	41·5	52·2
Yorkshire .	8	41·6	52·5
Derbyshire	2	41·0	53·6
		Cake Charges.	
Durham .	1	32·0	41·0
S. Yorkshire	6	36·8	47·5
Derbyshire	9	35·1	48·9

If the total porosity of a coke falls below about 50 per cent. it may not burn satisfactorily. For example, a Lancashire foundry coke made in beehive ovens, having a total porosity of only 43–45 per cent., was unsatisfactory in a foundry cupola, whereas coke made from the same coal cake-charged to a by-product oven had a total porosity of 50 per cent. and was a good foundry coke. In comparative "combustibility" tests in a 1-ft. diameter furnace, burning 39 lb. of coke per sq. ft. per hr., the maximum temperature of combustion of the beehive coke was only 1640° C., whereas that of the by-product oven coke was 1680° C.*

* See "Coke for Blast Furnaces," Part I, Chap. VIII.

Experiments by A. Killing (*Stahl und Eisen*, 1931, **51**, 901; *Fuel in Science and Practice*, 1932, **11**, 441) throw light on this subject. Cokes of different porosity were made from the same coal by grinding it in different ways so as to alter the bulk-density of the charge in the oven. The permeability to gases of 1-cm. cubes of the cokes, under a pressure of 10 cm. w.g., was then determined, with the results recorded in Table XXXIV.

TABLE XXXIV.—THE POROSITY OF COKE AND ITS PERMEABILITY TO GASES.

Per cent. of oven charge through ⅛-in.	Per cent. Pores in Coke	Permeability, c.c. of carbon dioxide per minute
59 (unground)	48·5	32
71 (passed once through disintegrator) .	50·5	42·7
83 (passed twice through disintegrator) .	53·4	51·9
92 (pulverised in hammer-mill) . .	55·2	154·0

Killing records that, as compared with the coke made from unground coal, the coke of highest porosity, prepared from coal pulverised so that over 90 per cent. passed through $\frac{1}{8}$-in., effected a saving in coke-consumption in a blast-furnace of over 2 cwt. per ton of pig-iron. It will be noted that the permeability of this coke to gases was remarkably high.

A comparison of the results recorded by G. A. Dummett and G. J. Greenfield (*Coke Oven Managers' Yearbook*, 1935, p. 129) for the bulk-density of coals of varying moisture-content (and size analysis), with those of G. A. Dummett and R. Stancey (*Coke Oven Managers' Year Book*, 1935, p. 268), for the impact hardness of cokes made from the same or similar slacks of varying moisture-content, shows maximum $1\frac{1}{2}$-in. shatter indices of the cokes and minimum bulk-densities of the coals for a range of moisture-content of 4 to 6 per cent. This suggests a parallel with Killing's results, for a charge of low bulk-density will yield a coke of high porosity, and the relationship traced by Killing between the porosity of a coke and its consumption per ton of pig in the blast-furnace may be due to the higher impact hardness of the coke of high porosity.

In tests now being made on the permeability of coke to the passage of gases it has not been found possible, for a wide range of cokes, to trace any general relationship between permeability and porosity.

Bulk Density

Killing had observed, in his experiments, that there was considerable difficulty in obtaining reliable values for the apparent density of coke from which to calculate its porosity. He was able, instead, to use bulk-density values, the measurements being simplified by the uniformly low moisture-contents (2 per cent.) of the cokes and their hardness, which caused them to yield but little smalls, the average size-analysis being, per cent.: over $4\frac{3}{4}$-in., 4; $4\frac{3}{4}$–2-in., 82; under 2-in., 14.

Early determinations of bulk-density were made by Killing with a vessel of 1 cu. m. capacity, but later a wagon of 28 cu. m. capacity was used. This wagon was loaded with coke through adjustable shoots to prevent breakage, taking equal amounts from each of three shoots from a bunker, to avoid differences in repeat tests due to the segregation of different sizes within the bunker. The values recorded in Table XXXV were obtained.

The relationship between the bulk-density of the coke made and its porosity was recorded over a period, as in Fig. 15. It was found that the coke-consumption at the blast-furnaces invariably fell with decrease in the bulk-density of the coke, *i.e.* with increase in its porosity.

TABLE XXXV.—RELATIONSHIP BETWEEN POROSITY AND BULK-DENSITY OF COKE. (Killing.)

Per cent. of Oven Charge through $\frac{1}{8}$-in.	Bulk-Density of Oven Charge, kg. per cu. m.	Bulk-Density of Coke, kg. per cu. m. (calculated to dry coke)	Porosity of Coke, per cent.
66 (unground) .	671·4	454	48·9
80 (ground) .	639·8	442	50·2
89 (finely ground)	595·8	429	52·4

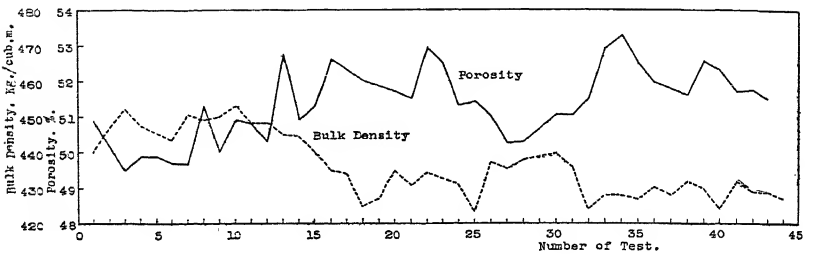


FIG. 15.—The Relationship between Bulk Density and Porosity of a Coke (Killing).

This method of measuring the average density of coke would be more difficult to use as a works test if much smaller coke were formed. The coke could be screened over or between specified screens, taking precautions against variation in ash and moisture content, against the segregation of sizes and against breakage during loading.

Bulk-density measurements of coke are sometimes required for purposes for which tests on a small scale are satisfactory. The British Standards Institution have therefore specified such a test, as follows* :

"A cubical measure having a capacity of exactly 2 cubic feet ($15\frac{1}{8}$-in. on each internal edge) shall be used.

Coke from a shatter test (large, small and dust) shall be dropped into the measure from a shovel not more than 2-in. above it, until the coke is level with the top, care being taken to prevent any undue voids, but without any shaking down.

The contents of the measure shall then be weighed. The weight divided by 2 will give the weight per cubic foot of coke. The moisture-content of the coke shall be determined and the bulk-density expressed in terms of dry coke.

All determinations shall be made in duplicate, and further

* Abstracted by permission from British Standard No. 496, The Sampling and Analysis of Coke, obtainable from the British Standards Institution, 28, Victoria Street, London, S.W. 1.

determinations shall be made if the difference in duplicate results exceeds 1 lb. per cubic foot."

This test may serve as a useful guide to the volume occupied by a specified weight of coke in a blast-furnace or a ship's hold. It is also of value for testing coke to be used in open grates, for a coke of low bulk-density attains "bright fire" conditions sooner than does a heavier coke of the same reactivity.

The bulk-density of coke is influenced by (1) the real density of the coke-substance with the ash, (2) the porosity and (3) the shape and range of size of the pieces, which affect the closeness of packing. When using a bulk-density test as a guide to the porosity of a coke, it is necessary to know also its real density.

Real Density

The method of determining the real density of coke described in our first report (Part I, Chap. V) was Greenwood and Cobb's modification of Anderson's method (see *J. Soc. Chem. Ind.*, 1922, **41**, 181*T*). This method was adopted with but slight alteration by the British Standards Institution (*B.S. No.* 496), together with the method standardised by the Northern Coke Research Committee.

These two methods and a third, devised in our laboratories, have been subjected to detailed examination.* The original paper may be referred to for a discussion of the work of other investigators, whilst the experimental study is here recorded.

When determining the real specific gravity of coke it is necessary to know the volume of a known weight of coke powdered sufficiently finely to break up all, or substantially all, closed pores. To obtain this volume by a liquid displacement method, it is necessary to obtain good wetting of the coke powder so that the liquid used is in intimate contact with the coke and has displaced any adsorbed gas films. Any mechanically-entrained air must be removed.

Apart from its wetting effect, an immersion liquid of low density has the advantage that any small proportion of the coke material which may be of low density is less likely to cause a scum. The formation of scum when using water has led to the deduction, which will be shown to be erroneous, that water is a poor wetting agent for coke.

Other properties of the liquids used when determining the volume of the coke are important. Since the experimental procedure is dependent on weighing, the volume of the liquid in the bottle, with and without the coke, has to be calculated from the density of the liquid. If both weighings (that of the bottle full of liquid and that of the bottle containing coke and liquid) are made at the same temperature, no complication arises. When boiling has been resorted to, cooling is required and, unless great care is taken, small

* "The Determination of the Real Specific Gravity of Coke," by J. Hiles and R. A. Mott, *Fuel in Science and Practice*, 1937, **16**, 64.

differences in temperature may occur. It follows that the least errors are possible with liquids which expand or contract but little with small changes in temperature.

The Effect of Differences of Temperature During a Determination.—In the method most often used, a specific gravity bottle of 50 c.c. capacity is weighed full of liquid, and, after a weighed quantity of powdered coke has been boiled for a period in a bottle one-third to half full of liquid, the bottle is cooled to near the original temperature, filled and re-weighed. The significance of small differences in temperature when the two weighings are made has not been fully appreciated, and errors may arise with some liquids if a difference of as little as 0·1° C. occurs. This may be demonstrated as follows:

Let W_1 = Weight of bottle plus liquid at t_1° C.
W_2 = Weight of bottle plus coke plus liquid at t_2° C.
W = Weight of dry coke.
W_B = Weight of bottle.
V_1 = Volume of bottle at t_1° C.
V_2 = Volume of bottle at t_2° C.
v = Volume of coke.
s = Density of coke.
d_1 = Density of liquid at t_1° C.
d_2 = Density of liquid at t_2° C.
k = Coefficient of cubical expansion of glass (0·000027 approximately).

Then
$$W_1 = W_B + V_1 d_1 \quad . \quad . \quad . \quad . \quad . \quad . \quad (1)$$
$$W_2 = W_B + (V_2 - v)d_2 + vs \quad . \quad . \quad . \quad . \quad . \quad (2)$$
$$W = vs.$$

(2) − (1) gives
$$W_2 - W_1 = (V_2 - v)\ d_2 + W - V_1 d_1$$
or
$$W_2 - W_1 - W = V_2 d_2 - V_1 d_1 - v d_2$$
$$= V_2 d_2 - V_1 d_1 - \frac{W}{s} d_2$$
or
$$s = \frac{W d_2}{W + W_1 - W_2 + V_2 d_2 - V_1 d_1}.$$

V_2, the volume of the bottle at t_2 ° C. $= V_1\ [1 + k\ (t_2 - t_1)]$.

Then
$$s = \frac{W d_2}{W + W_1 - W_2 + V_1 d_2 + V_1 d_2 k\,(t_2 - t_1) - V_1 d_1}$$
$$= \frac{W d_2}{W + W_1 - W_2 - V_1\,(d_1 - d_2) + V_1 d_2 k (t_2 - t_1)} \quad . \quad . \quad (3)$$

If $t_1 = t_2 = 15$° C.; and $d_1 = d_2 = d_{15}$
$$s = \frac{W d_{15}}{W + W_1 - W'_2}$$

W'_2 being the weight of bottle, plus coke, plus liquid, at 15° C.

Since the term $V_1 (d_1 - d_2)$ is always greater than $V_1 d_2 k (t_2 - t_1)$, the expression

$$\{ -V_1(d_1 - d_2) + V_1 d_2 k (t_2 - t_1) \}$$

is always negative. If this expression is omitted from the denominator in equation (3) the uncorrected value for s is low. It may be shown (see Appendix II) that when putting $W = 2$, for a coke of sp.gr. $= 2 \cdot 000$, the correction to be added when $t_2 = 16°$ C. and $t_1 = 15°$ C. is equal to:

$$\frac{2(X - Y)}{X - Y + d_{16}} \quad . \quad . \quad . \quad . \quad . \quad . \quad (4)$$

where $X = V_1 (d_1 - d_2)$ and

$$Y = V_1 d_2 k (t_2 - t_1) = V_1 d_2 k.$$

The values for X and Y for different liquids, when $t_1 = 15°$ C., $t_2 = 16°$ C., are recorded in Table XXXVI with the corrections for expression (4).

TABLE XXXVI.—ERROR $\frac{2(X-Y)}{X-Y+d_{16}}$ IN REAL SPECIFIC GRAVITY OF COKE FOR 1° C. INCREASE IN TEMPERATURE DURING A TEST USING DIFFERENT LIQUIDS IN A SPECIFIC GRAVITY BOTTLE.

Liquid	Density (d_{16})	X	Y	X − Y	$\frac{2(X-Y)}{X-Y+d_{16}}$
Carbon tetrachloride	1·60186	0·0960	0·0021	0·0939	0·111
Carbon disulphide	1·26845	0·0740	0·0016	0·0724	0·108
Water	0·99894	0·0090	0·0013	0·0077	0·015
Benzene	0·88307	0·0530	0·0011	0·0519	0·111
Toluene	0·86937	0·0465	0·0011	0·0454	0·099
Ethyl alcohol	0·79274	0·0425	0·0010	0·0415	0·099
Ethyl ether	0·71827	0·0565	0·0009	0·0556	0·144

It is noteworthy that all the liquids except water give a serious error for a temperature difference of 1° C., because of their much greater thermal expansion. The error is also influenced by the actual density of the liquid, carbon tetrachloride giving the greatest change of density for 1° C. difference, but its high density neutralising the effect on the results; the high change of density of ether for 1° C. difference, in conjunction with its low density, makes it most liable to cause error.

To obtain an accuracy of 0·005 in a real specific gravity determination, it is therefore necessary to keep the temperature constant to 0·03° C. for ether, and to about 0·05° C. for the other liquids, except water, which requires the temperature to be correct to 0·33° C. The magnitude of the error introduced by this factor has probably not been appreciated fully in the past, and the use of insufficiently sensitive thermostats when using organic liquids has probably caused many errors in results.

Water is outstanding in its low thermal expansion, but this increases with increase in temperature, the correction to the real specific gravity being 0·018 for a rise of temperature of 24 to 25° C. compared with 0·016 for the range 15 to 16° C. It is clear, however, that if the temperature of the liquid in the bottle when the two weighings are made does not differ by more than 0·25° C., no serious error will be introduced. Even when using water as the liquid, thermostatically-controlled baths should be fitted with a stirrer, and should be lagged to reduce the loss of heat. When using other liquids, much more careful temperature-control is necessary.

A Comparison of Different Liquids for Use in a Determination of Real Specific Gravity.—Certain properties of liquids, which have been used in the determination of real specific gravity of coke, are recorded in Table XXXVII.

TABLE XXXVII.—PROPERTIES OF LIQUIDS USED IN SPECIFIC GRAVITY DETERMINATIONS.*

Liquid	Density, d_{15}	Decrease in Density per 5° C. rise	Boiling Point, ° C.	Fluidity, rhes (at 25° C.)
Carbon tetrachloride	1·60378	0·00959	77	113
Carbon disulphide	1·26993	0·00742	46	290
Water	0·99912	0·00089	100	112
Benzene	0·88409	0·00532	80	165
Toluene	0·87036	0·00465	111	181
Ethyl alcohol	0·79357	0·00428	78	92
Ethyl ether	0·71940	0·00563	35	448

* International Critical Tables.

The fluidity is the inverse of the viscosity. Ether was used by Oshima and Fukuda (*Journ. Faculty of Eng., Tokyo Univ.*, 1929, **18**, 134), because it is one of the most penetrative liquids. Taking the fluidity as a measure of penetrativeness, water is one of the least effective of these liquids, but the value of the penetrativeness of the liquid must be questioned for use with coke. Oshima and Fukuda were mainly concerned with wood charcoal, the porous structure of which is much finer than that of coke. So long as the size of coke used is such that all closed pores have been broken open, there is no value in using a liquid of unusual penetrative power.

Burrows Moore (*Fuel in Science and Practice*, 1931, **10**, 442) has shown that coke of 200-mesh size has a greater absorptive power for water than for benzene or paraffin hydrocarbons. Since adsorption must precede absorption and adsorption is related to wetting, this may be taken to indicate a readier wettability of coke by water than by hydrocarbons. This conflicts with the experience of many operators making determinations of real specific gravity

with water, when scum is usually formed. Since scum is not formed when using organic liquids, it has been assumed that coke is less readily wetted by water than by organic liquids. The explanation is probably that no coke is homogeneous, and that most cokes contain a small proportion of material incompletely carbonised and therefore richer in hydrogen and volatile matter than the average.* This portion of the coke is less readily wetted by water than by liquid hydrocarbons. There is no doubt also a density

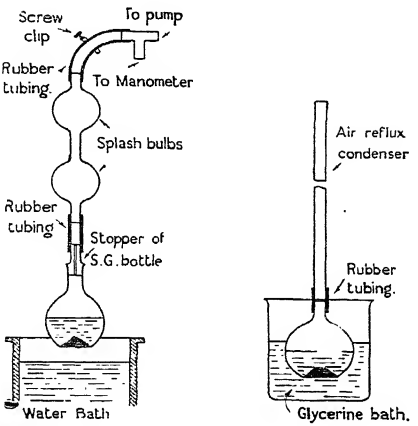


FIG. 16. FIG. 17.

Apparatus for the Determination of the Real Specific Gravity of Coke.

effect, the scum being lighter than water, but heavier than all but two of the organic liquids listed in Table XXXVII. With organic liquids, even oxygenated compounds like alcohol and ether, no scum is formed on the surface, but even if the wettability by the liquids of that portion of coke which gives a scum with water is less than with water, their lower densities would allow the scum to sink, and therefore simplify the procedure for determining the real specific gravity.

The Effect of the Size of the Coke.—The first standard method, in which the coke is boiled with water under reduced pressure, is conveniently referred to as method I (Fig. 16), and the second standard method, boiling in a glycerine-water bath with a reflux condenser, is referred to as method II (Fig. 17). Both methods were used for an investigation of the effect of size of coke on the value obtained for the real specific gravity, but only those obtained

* The flotation of a coke of 60-mesh size in liquids of different density showed that 10 per cent. was under 1·70 sp. gr., 75 per cent. of 1·70 to 1·77 sp. gr., and 15 per cent. over 1·77 sp. gr.

by method II, which will later be shown to be the more accurate, are here recorded. Air-free water was used with two sizes of coke—one in which the coke was crushed to pass 60-mesh I.M.M. (72-mesh B.S. test sieve); and the other, in which half of the original sample was ground in an agate mortar to pass a 200-mesh I.M.M. sieve. Six cokes were tested and the results are recorded in Table XXXVIII. Before each weighing the specific gravity bottles were immersed in a thermostatically-controlled bath with a stirrer, at a temperature 25° C.

TABLE XXXVIII.—THE EFFECT OF SIZE OF COKE ON THE VALUE FOR REAL SPECIFIC GRAVITY.

No.	Description	60-mesh "coke"		200-mesh "coke"		Difference
		Specific Gravity	Average Deviation from Mean. (Two tests)	Specific Gravity	Average Deviation from Mean. (Two tests)	
17C*	South Yorks.	1·831	0·003	1·858	0·012	+0·027
27A*	South Yorks.	1·863	0·003	1·862	0·004	−0·001
31B	South Yorks. beehive	1·929	0·002	1·927	0·001	−0·002
1A	South Wales	1·948	0·000	1·961	0·015	+0·013
55	Ceylon graphite	2·245	0·008	2·222	0·009	−0·023
55	Wood charcoal	1·452	0·000	1·456	0·001	+0·004

* Cokes No. 17C and 27A were both made from Parkgate hards (mainly durain), No. 17C from crushed coal carbonised in a by-product oven and No. 27A from lump coal coked in a beehive oven. They were included because microphotographs of the structure suggested that there were more micropores in these cokes than in ordinary cokes, and the effect of unruptured pores with coarser grinding would be more obvious with them.

For those materials for which the difference exceeds 0·01, the average deviation from the mean is excessive for 200-mesh material and is high in one case (No. 55) for 60-mesh material. The differences are not significant. Even wood charcoal shows no advantage for the finer crushing. Since there would have been a consistent increase in the real specific gravity if crushing to 200-mesh ruptured pores which were closed when the size was only 60-mesh, it may be concluded that there is no advantage to be gained by crushing coke as finely as 200-mesh for the determination of real specific gravity.

The Effect of Incomplete Wetting of the Coke in Giving Low Results for Real Specific Gravity.—There is incomplete wetting of the coke when air in the specific gravity bottle is not completely removed during the boiling operation, and complete contact of the liquid with the coke is prevented by a film or a bubble of air. It

is possible also that gases occluded in the coke could effect a similar action, but a test in which 2 grammes of powdered coke in a dry specific gravity bottle were exhausted by means of a Sprengel pump and the gas removed was collected and measured, showed that the volume of gas corresponded exactly with the volume of the free space in the bottle and its connexions. It was concluded, since the pressure was reduced to about 2mm. of mercury during the test, that it was justifiable to assume that in boiling tests no release of occluded gas to produce a film over the surfaces of the particles would be likely.

The effect of air-films in causing incomplete wetting was studied by using method I (boiling with water under reduced pressure) and method II (boiling with water under a reflux condenser), using water which in one case was recently boiled and therefore air-free, and in the other was first saturated with air by bubbling. Six tests were made on each coke with the two variants of each method; the results are recorded in Table XXXIX. A thermostatically-controlled bath was used to ensure that the temperatures when the bottles were filled with liquid, before and after adding the coke, were the same.

TABLE XXXIX.—THE EFFECT OF AIR IN CAUSING INCOMPLETE WETTING.

Coke No.	3 B.		13 J (Retort Carbon)		18 B.	
	Mean Sp. Gr.	Average Deviation	Mean Sp. Gr.	Average Deviation	Mean Sp. Gr.	Average Deviation
Method I						
Air-free water	1·940	0·016	2·072	0·017	1·894	0·011
Air-saturated water	1·935	0·015	2·098	0·020	1·884	0·011
Method II						
Air-free water	1·939	0·010	2·102	0·015	1·896	0·010
Air-saturated water	1·929	0·013	2·077	0·010	1·886	0·005

The results with air-saturated water were, with one exception, consistently lower, though the differences were usually small. Method II (boiling under a reflux condenser in a glycerine-and-water bath) gave the lower average deviation, and slightly higher results, due, no doubt, to the fact that the boiling at atmospheric pressure is more vigorous than under reduced pressure, and effects more complete removal of air mechanically entangled between the particles of coke at the beginning of a test.

It is concluded that air on the surface of the coke particles may cause low results, and that the use of air-free distilled water is desirable. The more vigorous boiling possible in method II is advantageous in removing air mechanically entrapped between

the coke particles, and this method was accepted in subsequent tests as being the more reliable.

The Effect of Adsorption in Giving High Results for Real Specific Gravity.—During the experimental work, on occasion, higher values for the real specific gravity of cokes were obtained than appeared to be justified. High results may be obtained when certain organic liquids are used, but the most abnormal results occurred when using a wetting agent with water. The use of a wetting agent was suggested as a means of overcoming the tendency for a small amount of scum to form on the surface of water in the specific gravity bottle, the loss of which in closing the bottle would give low results. A wetting agent produced by Messrs. Imperial Chemical Industries, Ltd.—namely, Perminal—has been used to lay coal dust in mines (Tideswell and Wheeler, *Trans. Inst. Min. Eng.*, 1933-34, **87**, 1). Its use in dilute solution in the ordinary boiling methods for determining real specific gravity was found to lead to frothing, and a modified method was devised which, at first, appeared to offer advantages over the other methods. The method consisted in adding 2 grammes of dry coke to a weighed specific gravity bottle, and then evacuating with a water or a Sprengel (mercury) pump. After applying suction for 30 minutes, distilled water, containing 1 per cent. of Perminal, was allowed to run into the bottle and the bottle was weighed. Since no heating took place, the time required to adjust the temperature of the bottle was reduced, and a saving in time was effected. A series of results using this method (method III) in comparison with method I are recorded in Table XL.

TABLE XL.—HIGH VALUES WITH METHOD III. COKE NO. 30B (DURHAM).

Method	Tests	Mean Sp. Gr.	Average Deviation	Remarks
I	8	1·989	0·003	Using air-saturated water.
III	4	2·163	0·081	Using air-saturated Perminal.
III	6	2·102	0·025	Using air-free Perminal.

Evacuation with a Sprengel pump allowed the collection and measurement of the air from the system. The gas consisted of air only and corresponded exactly with the volume of the system not occupied by the coke, so that no occluded gases were withdrawn. The high average deviations for method III show the greater difficulty in duplicating results with this method, particularly when the Perminal was saturated with air. Accepting the value obtained by method I as being correct, the high values by method III could only arise from the fact that W_2 (the weight of bottle + coke

and liquid) was greater than in method I, and that, therefore, more liquid entered the bottle in method III. This could only be accounted for by assuming, either that, in method I, all the entrapped air was not removed, or that, in method III, a condensed film of liquid occurred at the surface of the coke particules (adsorption) or that liquid penetrated into the particles (absorption). Since it has been shown that all closed pores were destroyed by grading to through 60-mesh, the formation of a condensed film was most probable.

Tests were therefore made on several cokes to see whether the experience was general. Since the adsorption seemed to be probably due to the Perminal, water was substituted for it in further tests by method III, and Perminal was substituted for water in method I. The results are recorded in Table XLI, those by method II being taken as standard.

TABLE XLI.—FURTHER TESTS ON THE FORMATION OF AN ADSORBED FILM.

Coke No.	3 B.		13 J.		18 B.		22 C.	
	Sp. Gr.	Average Deviation	Sp. Gr.	Average Deviation	Sp. Gr.	Average Deviation	Sp. Gr.	Average Deviation
Method II .	1·939	0·010	2·102	0·015	1·896	0·010	1·844	—
Method III .	1·962	0·010	2·266	0·002	1·906	0·002	1·880	0·007
Method III*a* .	—	—	2·095	—	—	—	1·863	—
Method III*b* .	—	—	2·040	—	—	—	1·682	—
Method I*a* .	2·024	0·009	2·177	0·002	1·921	0·004	1·902	0·005

Method III . Water pump evacuation of bottle containing dry coke, followed by admission of air-free Perminal water.
Method III (*a*) Water pump evacuation of bottle containing dry coke, followed by admission of air-free water.
Method III (*b*) Water pump evacuation of bottle containing dry coke, followed by admission of air-saturated water.
Method (*a*) . Boiling with air-free 1 per cent. Perminal under reduced pressure.
Method II . Boiling with air-free water with a reflux condenser.

In all cases, method III gives high results, but the values are reduced, approximately to the "correct" results, when air-free water replaces 1 per cent. Perminal; the results fall below the correct figures when the air-free Perminal is replaced by air-saturated water. These results were checked by making six determinations on three of the cokes by various methods. The results are reported in Table XLII.

It is clear that the use of air-free Perminal leads to high results when methods II or III are used, and the average deviations are also high. It is concluded that the use of 1 per cent. Perminal may lead to results which are abnormally high because of the formation of an adsorbed layer.

TABLE XLII.—MEAN VALUES OF SIX DETERMINATIONS BY VARIOUS METHODS.

	3 B.		13 J.		18 B.	
	Sp. Gr.	Av. Deviation	Sp. Gr.	Av. Deviation	Sp. Gr.	Av. Deviation
Method II						
Air-free water . . .	1·939	0·010	2·102	0·015	1·896	0·010
Method III						
Air-free 1 per cent. Perminal	1·966	0·034	2·113	0·042	1·946	0·027
Method III (c)						
Air-saturated 1 per cent. Perminal . . .	1·922	0·016	2·041	0·029	1·861	0·021
Method II (a)						
Air-free 1 per cent. Perminal	2·017	0·043	2·185	0·021	1·961	0·021
Method I (a)						
Air-free 1 per cent. Perminal	1·920	0·012	2·096	0·030	1·892	0·021

It was considered probable that certain organic liquids would be liable to give a condensed layer on the surface, and tests were made with various liquids. Method II was used, and coke of through 60-mesh, and the air condenser was replaced by a water-cooled condenser because of the low boiling-point of some of the liquids; whilst removable caps were slipped over the stoppers to prevent evaporation during weighing. The tests were carried out at the boiling-point of the liquid and the bottles were placed in a thermostatically-controlled bath at 25° C. for 30 minutes before each weighing. The results are recorded in Table XLIII.

TABLE XLIII.—THE USE OF ORGANIC LIQUIDS IN DETERMINING THE SPECIFIC GRAVITY OF COKE.

Liquid	Mean Sp. Gr.	Average Deviation *	Remarks
Air-free water . . .	1·985	0·007	—
Benzene	2·054	0·028	—
Carbon tetrachloride . . .	2·086	0·031	—
Toluene	2·148	0·008	Vigorous boiling.
Toluene	2·060	0·044	Gentle boiling.

* Two tests.

The use of organic liquids leads to high results with high average deviations and the formation of a condensed layer is again suggested. In these tests the organic liquids were not boiled free from air before the tests. The results might have been still higher with air-freed liquids.

In tests made by method III, using alcohol, carbon tetrachloride and carbon disulphide in place of 1 per cent. Perminal, values of 1·982 ± 0·027, 1·891 ± 0·006 and 1·941 ± 0·002 were obtained,

the value with 1 per cent. Perminal being $1{\cdot}841 \pm 0{\cdot}002$, which, in this instance, agreed well with the value found by method I.

Conclusions.—(1) It is apparent that the use of either organic liquids or wetting agents with water is liable to give abnormally high results for the real specific gravity of coke, by the formation of a condensed layer of liquid on the solid surface. Since organic liquids in general have a much higher coefficient of expansion than water, and require much more careful temperature-control, it is concluded that their use is not to be recommended, and that the higher results obtained with their use are misleading. Water is not liable to give a condensed layer on the surface of coke, and, since its coefficient of expansion is low, it is the most suitable liquid to use.

(2) All closed pores are opened by crushing coke to through 60-mesh, and there is no necessity to use coke more finely ground than this.

(3) Air is readily adsorbed on the surface of coke, and the liquid used for the determination of the specific gravity of coke should be freshly boiled before use.

(4) Vigorous boiling is helpful in displacing air in the specific gravity bottle, and the use of a glycerine-water bath to enable the water in the specific gravity bottle to boil at 100° C. under a reflux condenser is advantageous and gives the most complete elimination of air. This method is preferable to method I, although the latter is one of those recommended by the B.S.I.

(5) Temperature-control is facilitated by the use of a stirred and thermostatically-controlled bath.

Method.—As a result of this work, the method now recommended for determining the real density of coke is as follows :

Clean a 50 c.c. specific gravity bottle with chromic acid, rinse and fill with air-free distilled water and immerse in a bath of water at a convenient temperature (about 15° C.) during 1 hr. Note the weight of the bottle exactly filled with distilled water at this temperature (W, at t_1 °C.). Empty the bottle, dry the neck, and add exactly 2 grm. of dry coke ground to pass a 72-mesh B.S. test sieve and 25 c.c. of air-free distilled water. Remove the entrained gas by boiling under an air (reflux) condenser on a glycerine-water bath for ½-hr. (see Fig. 17). After half an hour wash down the scum with a few c.c. of hot air-free distilled water. Allow the bottle to stand for 1 hr. in a bath of water at t_2°C. (about 15° C.), then fill with air-free distilled water at the same temperature as the bath and weigh (W_2).

If t_1 and t_2 are equal the true specific gravity

$$= \frac{W}{W + W_1 - W_2}$$

where W = weight of dry coke (2·000 grm.).

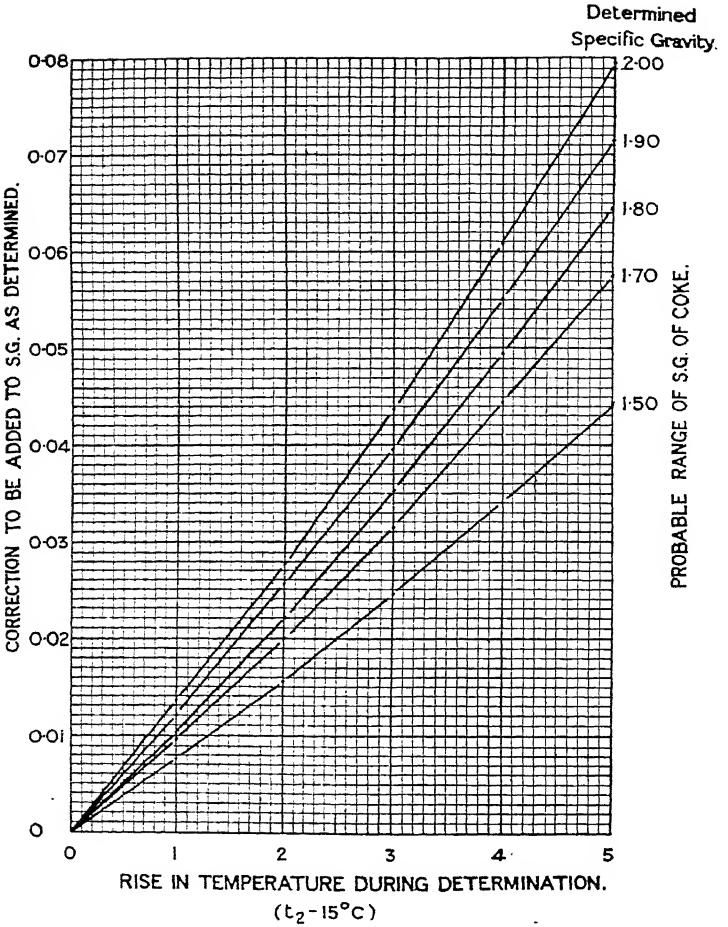


FIG. 18.—Chart for Correcting Real Specific Gravity for Changes of Temperature during a Determination.

Precautions may be taken, for example, by using a thermostatically-controlled bath, or by the use of cooling media, so that the bath temperatures t_1 and t_2 are the same, but if this cannot conveniently be ensured, a correction for the difference in temperature is made according to Fig. 18. The basis for this chart is the use of a 50 c.c. specific gravity bottle, with t_1=15° C. and t_2 higher. The correction read from the chart for the increase in temperature (t_2—15° C.) is then added to the determined value. The correction

is approximately 0·012 for each 1° C. rise above 15° C. for high-temperature cokes. The chart has been based on the following formula, which may be used for the calculation of the true specific gravity determined under other conditions:

$$\text{True specific gravity} = \frac{W(d_2)}{(W+W_1-W_2)-V(d_1-d_2) \cdot Vk(t_2-t_1)d_2}$$

where W = weight of dry coke (2·000 grm.

W_1 = weight of bottle plus water at t_1° C. (15° C.).

W_2 = weight of bottle plus coke plus water at t_2° C.

V = volume of specific gravity bottle in c.c.

d_1 and d_2 are the densities of water at t_1° and t_2° C. respectively.

k = the coefficient of cubical expansion of glass (0·000027).

All determinations shall be made in duplicate and further determinations shall be made if the difference in duplicate tests exceeds 0·01.

Calculation of Real Density of Coke on an Ash-free Basis.—To calculate the real density of the coke-substance free from ash, the following formula should be used. The specific gravity of coke-ash is assumed to be 2·5.

Real density on ash-free basis $= \frac{2{\cdot}5d\ (100-a)}{250-ad}$, d being the real density as determined, and a the amount of ash per cent.

For cokes containing less than 12 per cent. of ash, this formula gives nearly the same values as the more usual $\frac{100d-2{\cdot}5a}{100-a}$, but it is the more accurate formula for cokes of higher ash-contents.

TABLE XLIV.—THE RANK OF COAL AND THE REAL DENSITY OF ITS COKE.

Coal	Carbon per cent. (dry, ash-free basis)	Swelling-Power, per cent.	Real Density of Coke (ash-free basis)
Two Foot Nine (S. Wales)	89	145	1·93
Victoria (Durham)	89	135	1·96
Busty (Durham)	88	129	1·80
No. 5 (Kent)	87	126	1·87
Halifax Soft (Yorks.)	86	136	1·91
Mountain Mine (Lancs.)	86	—	1·87
Silkstone (Yorks.)	86	100	1·91
Parkgate (Yorks.)	85	127	1·83
Barnsley R. (Yorks.)	84	65	1·75
Winter (Yorks.)	83	8	1·71
Barnsley H. (Yorks.)	83	nil	1·71
Swallow Wood (Yorks.)	82	nil	1·71
Black Shale (Derby.)	82	12	1·78
Tupton (Derby.)	82	13	1·80
Deep Soft (Derby.)	82	19	1·71
Waterloo (Derby.)	81	nil	1·70

The Significance of the Real Density of Coke

Cokes made from good coking coals are of higher real density, up to about 2·0, than those made from inferior coking coals, which average about 1·7. Cokes made by blending a coking coal with a non-coking material, such as anthracite, are usually of low real density. The real density can serve as a rough guide to the reactivity of a coke, for those of low real density are the most reactive.

The relationship between the rank of a coal and the real density of the coke made from it is indicated in Table XLIV.

APPENDIX I

The Determination of the Density and Porosity of Small Specimens of Coke

It may be necessary to determine the density and porosity of a small sample of laboratory coke (see p. 307) in which the problem of selection of an average sample does not arise. The following method was devised for this purpose.

The greatest source of error in determining the apparent specific gravity of small pieces of coke is due to the large surface in relation to the volume, and the use of the usual methods would give a large amount of water entering the open pores. Although a correction may be applied for the water absorbed by weighing the coke after immersion in water, it is necessary to drain the coke to remove excessive surface water and water from open pores may also be removed. The least difficulty is introduced by filling the open pores completely with water and removing the excess surface water without draining.

The cokes described on p. 307 were added to a specific gravity bottle of 75 cc. capacity with a wide bore neck (1-in.) fitted with a capillary stopper. It is preferable to have the coke in one piece to keep the external surface small, but, if not, pieces as large as possible are added to half-fill the bottle. The stopper is inserted and connexion made through a T-piece, on the one side, to a supply of air-free distilled water in a suitable flask (into which the tube connected with the stopper dips) and, on the other side, to a mercury gauge and a water pump. On either side of the T-piece is a tap. By opening both taps the tube from the water supply to the first tap is filled with water and the tap is closed. Suction is applied to the coke in the bottle until the pressure drops to 20 mm. and this pressure is maintained for a short time. The tap near the water-pump is closed and the other opened to allow water to enter the bottle and fill it and the open pores of the coke. The determination is then completed as for the real specific gravity of coke and gives the weight of a volume of water equal to the volume of the cell walls and the closed pores.

The coke is removed from the bottle, the excess surface water is removed with filter paper, and its volume (that is the volume of the cell walls, closed pores and open pores filled with water, and therefore the apparent volume of the original coke) is determined in a suitable volumenometer by water-displacement.

These two determinations give the apparent volume of the coke and the volume of the cell walls and closed pores. By crushing the coke to pass 60 mesh the real density may be determined and the percentage of open and closed pores calculated.

APPENDIX II

THE EVALUATION OF THE CORRECTION TO DETERMINED REAL SPECIFIC GRAVITY WHEN $t_1 = 15°$ C. AND $t_2 = 16°$ C.

The specific gravity of coke is given by the formula

$$s = \frac{W d_2}{W + W_1 - W_2 - V_1 (d_1 - d_2) + V_1 d_2 k (t_2 - t_1)} \quad . \quad . \quad (4)$$

When $t_1 = 15°$ C., $t_2 = 16°$C.,

$$s = \frac{W d_2}{W + W_1 - W_2 - V_1 (d_1 - d_2) + V_1 d_2 k}$$

$$= \frac{W d_2}{W + W_1 - W_2 - X + Y} \quad . \quad . \quad . \quad . \quad . \quad . \quad (5)$$

where $X = V_1 (d_1 - d_2)$ and $Y = V_1 d_2 k$

If $t_1 = t_2 = 15°$ C.,

$$s = \frac{W d_{15}}{W + W_1 - W'_2} \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad (6)$$

(W'_2 being the weight of bottle, plus coke, plus liquid, at 15° C.)

The uncorrected value s_1 determined when $t = 16°$ C., is :

$$s_1 = \frac{W d_{16}}{W + W_1 - W_2}$$

$$\text{The correction } s - s_1 = \frac{W d_{15}}{W + W_1 - W'} - \frac{W d_{16}}{W + W_1 - W_2}$$

$$\text{or } W + W_1 - W_2 = \frac{W d_{16} (W + W_1 - W'_2)}{W d_{15} - (s - s_1) (W + W_1 - W'_2)} \quad . \quad (7)$$

$$\text{Also } \frac{s}{s_1} = \frac{W d_{16}}{W + W_1 - W_2 - X + Y} \div \frac{W d_{16}}{W + W_1 - W_2}$$

$$\text{or } s (W + W_1 - W_2 - X + Y) = s_1 (W + W_1 - W_2).$$

$$\text{Simplifying, } s - s_1 = \frac{s (X - Y)}{W + W_1 - W_2}.$$

Substituting the value of $(W + W_1 - W_2)$ from equation (7),

$$s - s_1 = \frac{s(X - Y) \{ W d_{15} - (s - s_1) (W + W_1 - W'_2) \}}{W d_{16} (W + W_1 - W'_2)}.$$

When $s = 2{\cdot}0$ and $W = 2$ grm., $W + W_1 - W'_2 = 1 d_{15}$, (from (6)).

$$2 - s_1 = \frac{2 (X - Y) \{ 2 d_{15} - (2 - s_1) d_{15} \}}{2 d_{16} d_{15}} = \frac{(X - Y) s_1}{d_{16}}$$

$$\text{or } 2 d_{16} - s_1 d_{16} = (X - Y) s_1$$

$$\text{or } s_1 = \frac{2 d_{16}}{X - Y + d_{16}}.$$

$$\text{The correction } 2 - s_1 = \frac{2(X - Y + d_{16}) - 2 d_{16}}{X - Y + d_{16}} = \frac{2 (X - Y)}{X - Y + d_{16}}.$$

CHAPTER IX

THE ABRADABILITY OF COKE

The Cochrane Test

Tests of the "abradability" of coke, the so-called "tumbler" or "drum" tests, appear to have been the earliest forms of physical tests made to assess its quality.

The drum test that has been most widely used in this country is that due to G. D. Cochrane (see *J. Iron and Steel Inst.*, 1918, **1**, 141) in which a charge of 28 lb. of coke of about 2–3-in. size is rotated in a drum 30 in. in diameter and 18 in. long during 55½ min. (1000 revs. at 18 r.p.m.) and screened on a ⅛-in. square mesh screen. The percentage remaining on the screen is used as an index of the "hardness" of the coke.

During 8 years' use of this test, Cochrane observed that the driving of a blast-furnace deteriorated if the "hardness" index of the coke fell below 74½ or 74. Whilst there seemed to be no advantage in using a coke with an index higher than 76, the rate of drive of the furnace decreased in proportion as the index fell below 74. Cochrane estimated that when using a coke of average index 70·5 during a year, the rate of drive of the furnace was reduced by 20 per cent.

The use of the term "hardness" by Cochrane may have led to some misunderstanding, but it is evident from his writings that he meant surface hardness or "abradability." For example (*loc. cit.*, p. 144), it is stated that a coke of low ash-content "although not hard is less friable than a coke containing a higher percentage of ash, owing to its not being so much split up by small pieces of stone." Moreover, Cochrane remarked that "a soft coke is usually very friable" and described an experience with a coke so soft and friable that the rate of drive of the blast furnace was reduced to such an extent that the slag would not run. The front tuyeres of the furnace were taken out and "great clouds of fine coke dust were blown out," after which the furnace drove freely.

Blast-furnace managers have no doubt attached importance to the surface hardness or abradability of coke, because serious abrasion only occurs within the blast-furnace itself, and may therefore be held to account directly for some of the vagaries of working of the furnace, whereas the liability of coke to breakage by impact (as measured by the "shatter" test) is incurred mainly

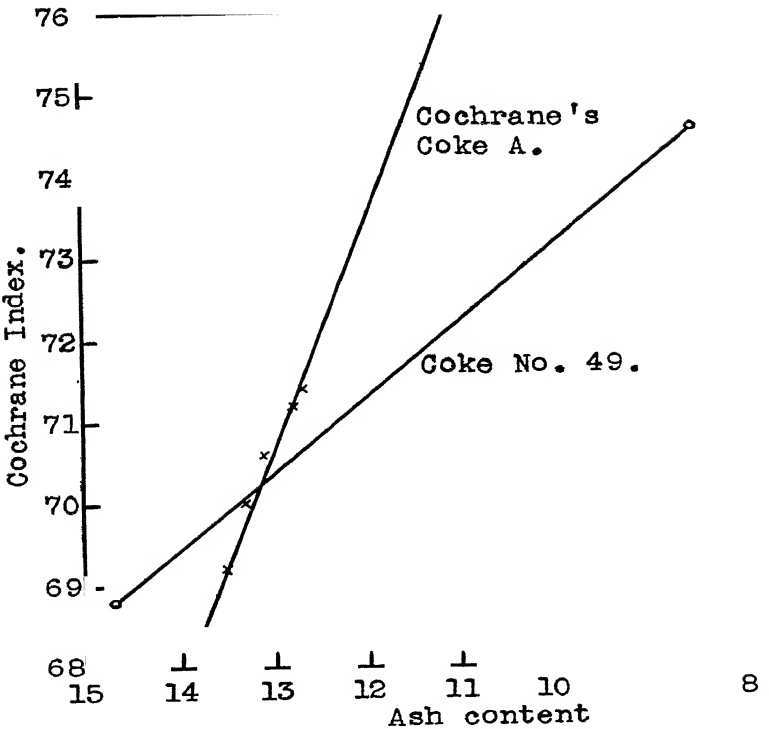


FIG. 19.—The Effect of Ash Content on the Cochrane Index of Two Cokes.

outside the blast-furnace. Cochrane has made it clear, however, that the abradability of a coke only became of importance in the working of his blast furnaces if its "index," as determined by his test, fell below 74. It will be shown shortly that this is an abnormally low value for present-day cokes.

An examination of Cochrane's records suggests that the abradability of his cokes (war-time cokes) was due mainly to their high ash-contents. His coke A, used for the majority of the tests, had an ash-content varying from 12 to over 16 per cent. From Fig. 2 of his paper (*loc. cit.*, p. 144) the following figures, representing average values over a number of days, can be deduced:

Ash-content of coke, per cent.	12·7	12·8	13·1	13·3	13·5
Cochrane index	71·4	71·2	70·6	70·0	69·2

The regularity of the decrease in the index with increase in the ash-content of the coke—plotted against each other the values

lie almost on a straight line (Fig. 19)—suggests that an index of over 76 would have been obtained had the coke contained no more than 10 or 11 per cent. of ash, and that such coke would not have adversely affected the drive of Cochrane's furnaces. Few present-day cokes contain more than 10 per cent. of ash, and it is reasonable to conclude that few normal present-day cokes have such a low Cochrane index as to affect the drive of a blast-furnace adversely.

Cochrane's coke A can be regarded as having been made from a blend with a "non-coking material" (*i.e.* the ash or shale), such as often results in the production of an abradable coke.

The abradability of a number of typical South Yorkshire cokes, as measured by Cochrane's test on delivery at an iron-works, is recorded in Table XLV.

TABLE XLV.—THE ABRADABILITY OF SOUTH YORKSHIRE COKES. (COCHRANE'S TEST.)

Coke No.	Ash-Content, per cent.	Shatter Indices		Cochrane Index ($\frac{1}{8}$-in.)
		$1\frac{1}{2}$-in.	$\frac{1}{2}$-in.	
*29	10·5	83	98·1	81·3
*HM	8·8	79	97·6	81·0
*65	8·0	74	97·8	80·1
*23B	9·6	80	98·1	79·9
*C	9·2	77	97·4	79·7
*71	10·4	84	98·2	79·5
70	9·4	—	97·7	77·5
*79	7·7	—	97·8	77·4
57	8·5	80	97·2	77·3
44	10·9	83	97·0	77·3
58	6·7	89	98·3	76·7
WS	8·1	77	97·1	76·7
3	9·7	83	97·5	76·3
13	8·0	83	97·5	76·2
63	10·4	85	97·8	75·9
7	8·1	82	97·3	75·8
80	10·2	78	97·0	74·7
W	7·8	86	97·9	74·0
3C (Beehive)	10·7	89	97·9	72·4
*BM	8·2	82	97·2	70·5
49	15·2	94	98·9	69·0

* Cake-charged.

Of the three cokes in this list having a Cochrane index of less than 74, No. 49 was made from unwashed coal, and contained 15·2 per cent. of ash, and No. 3C was a beehive coke of high porosity (60 per cent.). The low value (70·5) for coke BM is difficult to explain, the more so because it was made from cake-charged coal. In general, we have found that cokes made from compressed charges, though more fissured than top-charged cokes, and therefore more easily broken by impact, are dense and of high surface hardness.

The values recorded in Table XLV, with the exception [illegible] bear out this experience.

The Effect of the Shale-Content of Coke on its Abradability.—Coke No. 49 of Table XLV is now made from cleaned coal, and an opportunity was taken to compare the abradability of the coke before and after its ash-content had been reduced, with the results recorded in Table XLVI.

TABLE XLVI.—EFFECT OF ASH-CONTENT OF COKE NO. 49 ON COCHRANE ABRASION INDEX.

	Ash, per cent.	Cochrane Index	Ash, per cent.	Cochrane Index
	15·2	69·0	10·1	75·8
	14·8	69·5	9·2	73·5
	14·8	68·9	8·5	75·0
	14·5	68·0	7·9	70·5
	14·4	68·8	7·8	74·9
			7·5	77·8
Mean .	14·7	68·		74·6

The effective cleaning of the coal used for Coke No. 49 influenced the abradability of the coke considerably, though not to the same extent as with Cochrane's Durham coke (see Fig. 19). The coal used for coke No. 49 was finely crushed (90 per cent. through $\frac{1}{8}$-in.), finer than is usual for making many Durham cokes. The coarser crushing of the coal used in making the cokes used by Cochrane may have exaggerated the effect of the high shale-content.

An opportunity arose to test a coke made in England from Chinese coal of high fixed-ash content. The coal contained 13·8 per cent. of ash, but the shale-content was only 1·2 per cent., the high ash being chiefly due to finely-divided mineral matter disseminated throughout the coal. The results were as follows:

Ash in coke, per cent.	17·7
Cochrane Index	80·2
Shatter Index $1\frac{1}{2}$-in.	81·8
Shatter Index $\frac{1}{2}$-in.	97·6

A high ash-content does not, therefore, necessarily render a coke abradable. Since, however, British coals used for coke-making are usually of low fixed-ash content, it may be expected that an ash-content in coke exceeding about 10 per cent., indicating the presence of coarse particles of shale, would usually affect the abradability adversely.

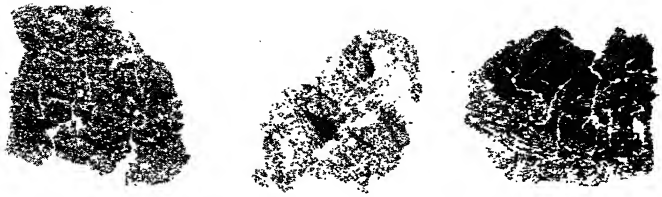
The Effect of the Structure of Coke on its Abradability.—An abradable coke is always produced if a non-coking or poorly-coking material is blended in too high proportion with a coking

FIG. 20.—" Pebbly " Structure of Coke due to Non-Coking Constituent.

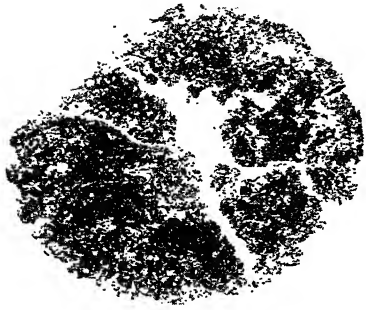
coal. This effect of such materials is due to the fact that they do not swell, or swell insufficiently, during coking. When an oven charge consists of a coking coal blended with a large proportion of a non-swelling or feebly-swelling material, there is imperfect agglomeration of the particles during coking due to lack of pressure, and the resultant coke may have a " pebbly " structure in which apparently unaltered particles of coal are visible. For example, slack from the Barnsley seam may contain from 10 to 50 per cent. of non-swelling durain, and there may be imperfect agglomeration during the coking of charges of such coal (unless the clarain and vitrain of the seam are strongly swelling) so that some of the coke has the pebbly structure illustrated in Fig. 20.

From coke with a pebbly structure, imperfectly-agglomerated particles can readily be pulled off with the fingers, and the fact that the coke is easily abradable is evident. Comparative tests made on lumps of coke with and without a pebbly structure, taken from the same charge, gave the following results :

	Shatter Indices		Cochrane Index
	1½-in.	½-in.	⅛-in.
Coke with pebbly structure . . .	75·6	94·5	67·2
Coke without pebbly structure . . .	73·2	96·4	76·9

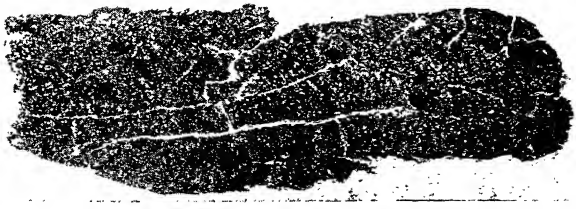


Vertical retort cokes



Coalite

Horizontal retort coke (N.W.)



Oven coke made from finely-crushed coal

FIG. 21.—Irregular Structure of Gas Cokes and Coalite compared with Uniform Structure of Oven Coke ($\times \frac{1}{3}$)

Despite its appearance, "pebbly" coke is fully carbonised. Under-carbonising also yields an abradable coke, whilst over-coking produces a coke with a high Cochrane index. Thus, in a battery of 16-in. ovens, normally coking in 14½ hours, cokes were made (charged at the same time) with 1½-hr. undercoking and 2-hr. over-coking. Tests of the cokes gave the following results :

	Shatter Indices				Cochrane Index, ⅛-in.
	2-in.	1½-in.	1-in.	½-in.	
Normal coke	55	73·6	87·4	96·1	74·9
Under-coked 1½-hr.	55	74·9	89·1	96·4	74·0
Over coked 2-hr.	54	73·0	89·0	95·6	78·8

Several gas cokes and low-temperature cokes have been tested with the following results :

	Shatter Indices		Cochrane Index, ⅛-in.
	1½-in.	½-in.	
Lancashire vertical retort coke	86·4	97·0	59·8
Yorkshire vertical retort coke	—	—	64·0
Yorkshire vertical retort Coke II	79·4	96·4	59·1
"Coalite"	81·6	95·4	59·1
"Natco" low temperature coke	55·5	88·5	65·7

The structures of the Lancashire and Yorkshire vertical retort cokes and of "Coalite" are illustrated in Fig. 21, and show irregularity with a considerable proportion of "sponge." For comparison, a horizontal retort coke made from rough coal, and an oven coke made from finely-crushed coal of low ash content are included in Fig. 21, the regularity of the structure of the oven coke being apparent. It will be observed that the Cochrane test distinguishes these "soft" cokes from ordinary metallurgical cokes.

The Effect of Blending Coking Slacks with Coke Dust on the Abradability of the Coke.—Coke dust may be added to a coking slack to increase the impact hardness of the coke. This practice is successfully applied at several coking-plants in South Wales, Durham, Lancashire and Yorkshire. If coke dust is added to a poor coking coal the coke may be made more abradable and, under such circumstances, the addition is harmful. Blast-furnace tests using a coke made under such conditions have been reported by W. J. Brooke, H. R. B. Walshaw and A. W. Lee (*Journ. I.S. Inst.*, 1936, **II**, 287). Coke breeze dried to 12 per cent. moisture-content was ground in a rod mill to pass through a 1 mm. screen and a 4 per cent. addition was made to Yorkshire coking slacks of

low rank over a period of 5 weeks. The coke-consumption at the blast-furnaces per ton of pig was, however, increased, the rate of drive fell off considerably and "slipping" increased. The most serious effect was on the rate of drive, one furnace carrying only 4,440 tons of burden in the fifth week of the test as compared with a normal 5,285 tons. Over the period of the trials, shatter tests of the coke were made with the following results:

	Shatter Index	
	1½-in.	½-in.
Five weeks before coke-dust blending	76·5	96·7
Five weeks during coke-dust blending	79·4	96·2

Cochrane abrasion indices were not made of the cokes at this time, but subsequent tests on coke made by blending with coke dust gave a mean value of 71·4. The average Cochrane index of the coke during the first complete five weeks after the tests (about one month after blending with coke dust was stopped) was 75·2, confirming Cochrane's deduction that cokes with abrasion indices under 74·0 influence the rate of drive of a blast-furnace adversely.

Whether or no blending with coke dust makes a coke abradable depends chiefly on the rank of the coal used and on the size of the coke dust added. The effect of these two factors will be discussed in a later chapter.

TABLE XLVII.—REPETITION TESTS ON COKE.

No. of test	Shatter Indices				Cochrane Index
	2-in.	1½-in.	1-in.	½-in.	
1	53·9	73·0	88·6	96·5	77·7
2	57·1	74·5	89·6	96·6	76·1
3	54·4	73·7	88·9	96·7	77·9
4	56·7	75·9	89·7	96·7	74·7
5	56·9	76·4	91·0	97·2	77·7
6	56·7	75·1	88·9	96·9	75·2
7	59·2	76·1	90·1	97·2	77·2
8	56·1	76·0	89·2	97·1	76·1
9	56·7	76·2	89·2	97·1	77·7
10	55·2	75·2	89·0	96·7	76·3
11	54·4	77·2	90·2	97·1	74·1
12	56·0	76·0	89·6	96·9	75·2
Mean . .		75·4		96·9	76·3
Maximum value		77·2		97·2	77·9
Minimum value		73·0		96·5	74·1
Average Deviation .		0·96		0·21	1·09

Accuracy of the Cochrane Test.—Messrs. John Lysaght, Ltd., have been good enough to communicate to us a record of special tests made to determine the variations in the shatter and Cochrane indices of coke made at their ovens. Over a period of 4 hours, about 2 tons of coke were collected by fork from a travelling belt carrying the coke from the ovens. This large sample was well mixed and one half of it taken for test by filling it into twelve sacks, each of which served for one abrasion test. This record of the tests is given in Table XLVII.

We have also had the opportunity of studying the results of daily routine rests (three per day) of the coke from the ovens at Messrs. John Lysaght, Ltd., and have calculated the average deviation for the ½-in. shatter index and the Cochrane index in 187 tests, as follows:

	Shatter Index, ½-in.	Cochrane Index, ⅛-in.
Mean of 187 tests	96·4	75·2
Maximum value	97·2	79·2
Minimum value	95·4	72·0
Average deviation	0·29	1·03

No doubt the coke varied in quality somewhat during the period of 10 weeks covered by the tests, but the variation would be small. It will be seen that the average deviations, both for the ½-in. shatter index and the Cochrane index, agree closely with the values given in Table XLVII for results obtained with carefully mixed and sampled coke.

Using the 187 routine tests, the accuracy of the Cochrane index has been compared with that of the ½-in. shatter index by the method of Briscoe and Marson (*Fuel in Science and Practice*, 1931, **10**, 464) in which the probable errors for means of groups of 3, 6, 9 . . . tests are determined. The probable error is that which has an even chance of occurring but, to ensure greater accuracy, it is necessary to know values for the errors that can occur, say, once in 11 times and once in 101 times. Such values have been graphed in Fig. 22 and from them the following examples can be considered:

	Errors for 10 : 1 Chance		Errors for 100 : 1 Chance	
	Cochrane Index, ⅛-in.	Shatter Index, ½-in.	Cochrane Index, ⅛-in.	Shatter Index, ½-in.
Single tests . . .	2·18	0·66	3·28	1·00
Average of 3 tests . .	1·24	0·36	1·87	0·55
Average of 6 tests . .	0·88	0·22	1·40	0·40
Average of 21 tests .	0·58	0·09	0·74	0·26
Average of 42 tests .	0·34	0·06	0·51	0·11

It will be seen that in a single test there is one chance in 100 of an error of 3·3 occurring in the Cochrane index or of an error of 1·0 occurring in the ½-in. shatter index; or, to put it another way, a single test is within 3·3 of the correct Cochrane index, or within 1·0 of the correct ½-in. shatter index in 99 out of 100 tests (a large proportion being much nearer the correct result).

During the period of the routine tests under consideration, the range of averages of the (three) daily tests was 76·9 to 73·1, or 3·8, for the Cochrane index; but an error of 1·87 can occur once

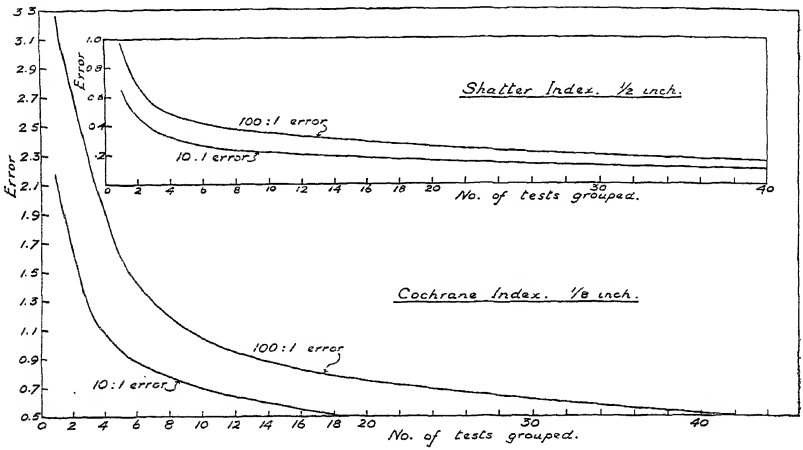
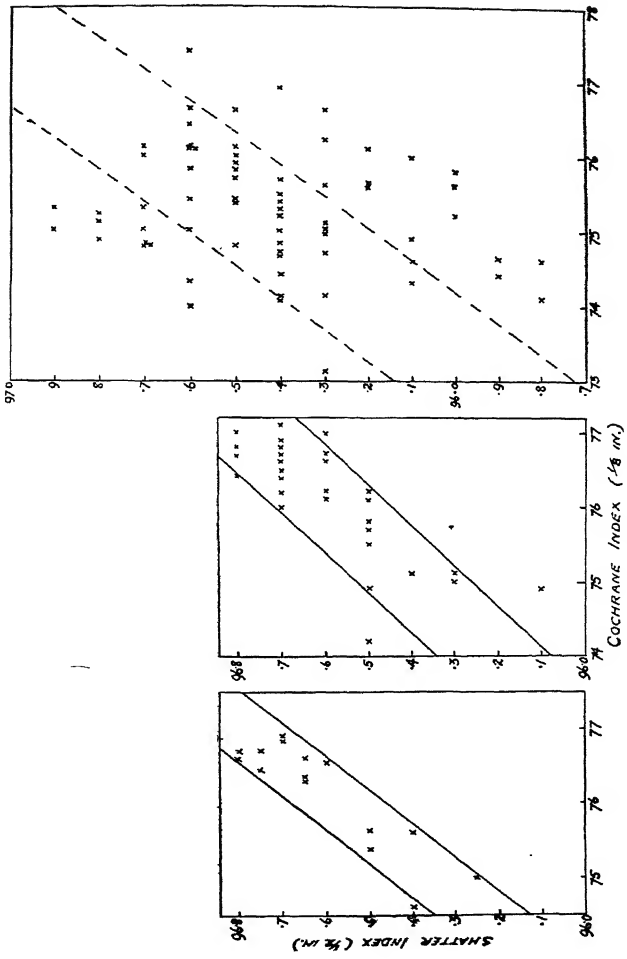


FIG. 22.—Errors in the Means of Groups of Tests, Cochrane Index and ½-in. Shatter Index.

in 100 times in the means when only three tests are made, so that such averages are not sufficiently accurate to be significant. If weekly averages are taken (21 tests) the error (100 : 1 chance) is reduced to 0·74. When averaging 21 tests for the ½-in. shatter index the error (100 : 1 chance) is 0·26. The Cochrane index is therefore more variable than the ½-in. shatter index.

Relationship between Cochrane Index and Shatter Index.—On the assumption that the errors calculated from the 187 tests to which Fig. 22 relates would be substantially the same over a longer period of testing, the weekly average results obtained by Messrs. John Lysaght, Ltd., during 28 weeks were studied. In Fig. 23b the weekly averages of the Cochrane indices and the ½-in. shatter indices are plotted against each other, and in Fig. 23a the fortnightly averages are so plotted; whilst, for comparison, the same treatment is given to the daily averages (3 tests) in Fig. 23c.

It will be seen that the magnitude of the errors involved in the



(a) Fortnightly. (b) Weekly. (c) Daily.

FIG. 23.—Comparison of Mean $\frac{1}{8}$-in. Shatter Index and Mean $\frac{1}{8}$-in. Cochrane Index. Values for Different Periods.

daily averages obscures any relationship that there may be between the two indices, whereas with the weekly and fortnightly averages the results lie within fairly narrow bands. From Fig. 23a (fortnightly averages) it will be seen that a coke having a Cochrane index of 75 may have a ½-in. shatter index between 96·25 and 96·45, a range of 0·2. On the other hand, a coke having a ½-in. shatter index of 96·5 may have a Cochrane index between 75·2 and 76·2,

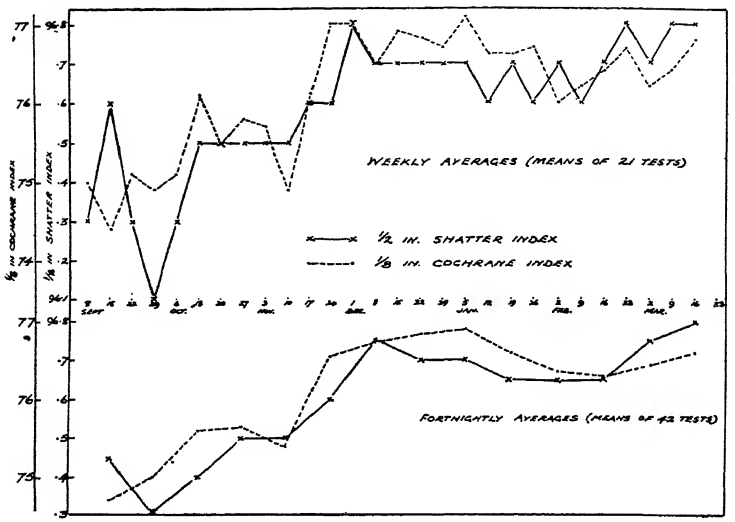


FIG. 24.—Comparison of Mean Values of ½-in. Shatter Index and ⅛-in. Cochrane Index for Period of Seven Months.

a range of 1·0. A range of 0·2 of the ½-in. shatter index is thus equivalent to a range of 1·0 of the Cochrane index.

In Fig. 24 the variation in the Cochrane indices and the ½-in. shatter indices are charted throughout the period studied, using scales such that a range of 1·0 for the Cochrane index is equivalent to a range of 0·2 for the ½-in. shatter index. The fortnightly averages show clearly that the indices for the most part rise or fall together, and are presumably, therefore, measuring either the same property of the coke or interdependent properties. Since the scales used in Fig. 24 for the two indices are in about the same ratio as the errors (100 : 1 chance) for the fortnightly averages, namely, 0·51 for the Cochrane index and 0·11 for the ½-in. shatter index, the two charts should keep the same distance apart if the same property is being measured by both indices. Actually, the charts cross or touch at 4 points, and it can be assumed that one or other, or both, of the tests may be influenced, on occasion, by some property of coke other than its surface hardness.

It is important to record that the coke with which this study was made was unusually uniform in character. The average deviation from the mean for fifty $1\frac{1}{2}$-in. shatter indices, determined over a period of 17 days, was 0·85 as compared with values of 1·08, 1·25 and 1·39 obtained in similar tests with Yorkshire cokes.

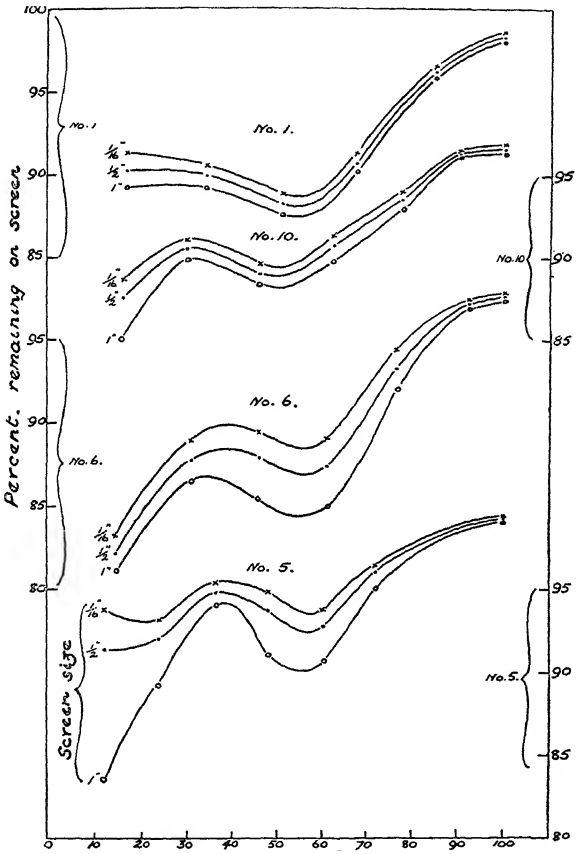


Fig. 25.—Percentages of different sizes of 4 Cokes remaining after Tests in Sheffield Abrasion Drum with Different Percentage Fillings.

The Sheffield Coke Abrasion Test

In the original Sheffield coke abrasion test, as described in "Coke for Blast Furnaces," p. 90, a drum test was modified in such a manner that the larger pieces of coke remaining at the conclusion of the test had a similar appearance to pieces raked from the tuyeres of a blast furnace, the inference being that the

abrasive treatment accorded the coke during the passage of the charge down the shaft of a blast-furnace was thus reproduced. The test should, therefore, give a good indication of the amount of dust thus formed from a coke in practice.

Briefly, the test involved the rotation of 2-cu. ft. of coke within a drum 18-in. long and 18-in. in diameter during ½-hr. at 23 r.p.m. The charge was then screened and the proportion remaining on 1½-in. was termed the "abrasion index." The most important departure from the procedure adopted in the Cochrane test was the use of a standard volume instead of a standard weight of coke. This change was made as the result of a series of experiments which showed that, if a standard weight were used, variations in the bulk-density of different cokes made it impossible to compare the effect of the test on them because the free space in the drum varied.

Fig. 25 illustrates the effect, when carrying out the tests, of varying the quantity of coke, and therefore the amount of free space in the drum. With the four cokes tested, most dust (through 1/16-in.) was formed when the drum was half-filled, whilst with three of them there was much dust with a 15 or 20 per cent. filling. The maximum production of dust with a 50 per cent. filling is no doubt due to the fact that the area of exposed surface of the charge of coke is then greatest. On the other hand, with a 15 or 20 per cent. filling the pieces of coke may be so free to move that all the surfaces become effective rolling surfaces, whilst increase in the filling above about 20 per cent. restricts the movement of many of the pieces below the exposed surface of the charge by reason of the superincumbent weight.

For all the cokes tested, the reduction in the amount of dust formed as the percentage filling was increased above 50 is marked. Thus, the amounts of through 1/16-in. coke produced with different percentage fillings were :

Coke No.	Percentage Filling of Drum					
	15	33	50	67	75	100
1	8·5	9·5	11·0	9·5	7·0	1·3
10	11·5	9·0	10·0	8·0	6·5	1·3
6	17·0	11·0	11·5	9·0	6·5	3·2
5	6·0	5·0	5·0	4·5	3·0	0·7

Except for coke No. 5, which was made from a cake-charge of Durham coal and was very dense, the order of abradability of the cokes, as indicated by the amount of through 1/16-in. dust formed, varies with the percentage filling up to about 50 per cent., the shape of the pieces of coke no doubt influencing the results.

It is thus clear that in such a drum abrasion test the percentage filling is one of the most important considerations, and that when

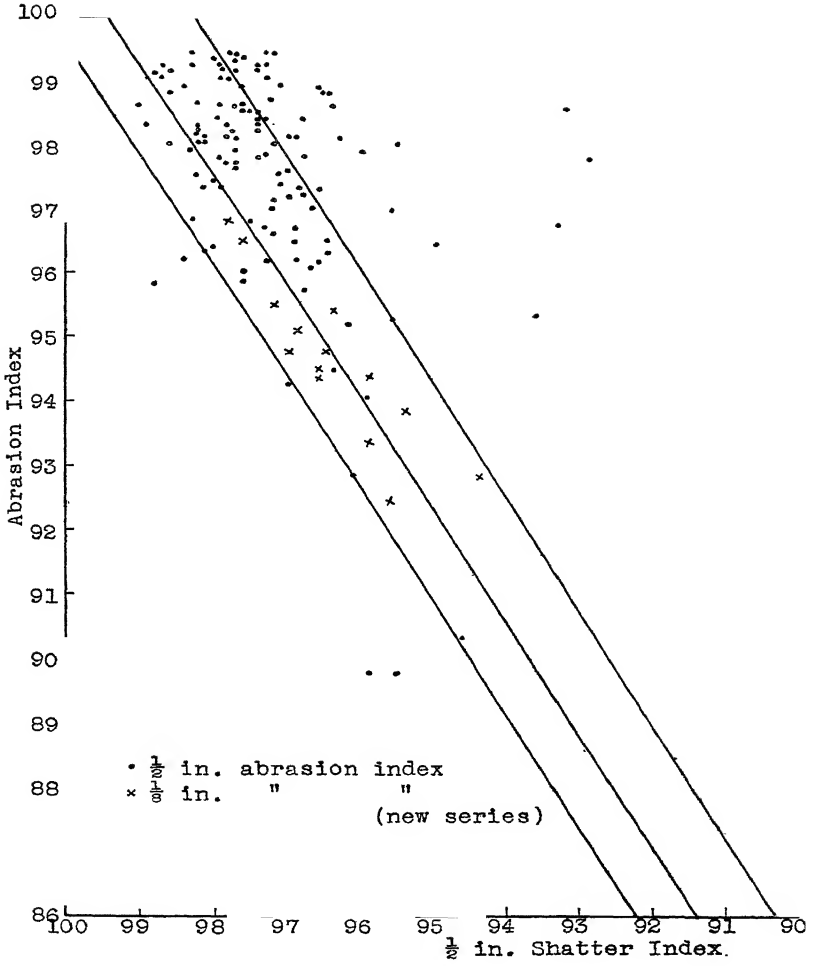


FIG. 26.—The Relationship between ½-in. Shatter Index and Sheffield Abrasion Index.

comparing by such a test cokes which differ considerably in bulk-density a standard volume rather than a standard weight of coke should be used. In a Cochrane drum, 30 in. in diameter and 18 in. long (capacity 7·35 cu. ft.), the charge of 28 lb. of coke might occupy from about 0·95 to 1·25 cu. ft. according to the bulk-density of the coke, the percentage filling thus varying between about 13 and 17 per cent. This variation is too small to affect the tests on robust

cokes, except possibly those of abnormally high or low bulk density.

The drum used in the Sheffield abrasion test was modified by the fitting of a liner of manganese steel, to enable it to be used as a ball or rod mill for grinding coke. This reduced its internal diameter to 17-in., its length to 15¼-in. and its volume to 2-cu.ft. We have since carried out a number of abrasion tests on experimental cokes (from our experimental oven) with this modified drum, using 1½-cu. ft. of coke (75 per cent. filling) rotated during 12½ min. at 60 r.p.m. These conditions of test produce similar results to those obtained with the original drum. The product is screened at ⅛-in., as in the Cochrane test.

In Fig. 26 the "⅛-in. abrasion indices" thus obtained for a number of cokes are compared with their ½-in. shatter indices. In the same diagram, "½-in. abrasion indices" as determined by the original Sheffield abrasion test are plotted against the ½-in. shatter indices of a number of cokes. The values, in general, fall into parallel bands, suggesting, as has been noted previously, that drum abrasion indices and the ½-in. shatter index measure the same property of coke. Particulars regarding the cokes which fall wide of the bands in Fig. 26 are :

Coke No.	Abrasion Index	Shatter Index (½-in.)	
3EI	97·8	92·9	Made from 1–½-in. uncrushed coal.
3EII	98·6	93·2	Made from ½–⅜-in. uncrushed coal.
3EIII	98·0	95·5	Made from ⅜–½-in. uncrushed coal.
13D (b)	96·8	93·3	Made from slurry.
42A	95·4	93·6	Beehive coke, Black Shale (Notts.) coal.
28A	97·0	95·6	Black Shale (Derby.) coal.
54A	96·5	95·0	Pitch coke.
29D(b)ii	97·9	96·0	Charge contained 2½% oxidised coal.
29I(b)v	89·8	95·9	Charge contained 30% low temp. coke breeze.
1A	95·8	98·8	Blend of S. Wales coking and steam coals.
	89·8	95·5	Charge contained 20% anthracite duff.

These are all abnormal experimental cokes.

A statistical study of tests on 250 different cokes has shown that only 51 had ½-in. shatter indices less than 97, 31 of them being less than 96·5. All except 6 of these cokes of low ½-in. shatter index were abradable cokes made from charges containing a proportion of non-swelling or poorly-swelling material. Of the 6 exceptions, 3 were made from uncrushed coal (cokes 3EI, II and III) and 3 were so heavily fissured that an unusual number of pieces less than ½-in. in size were broken off them by impact. In general, therefore, cokes for which the ½-in. shatter index falls below 97·0 may be found to be "abradable."

APPENDIX

Specification for the Drum Abrasion Test.*

(This test is under consideration for adoption as a British Standard)

The Cochrane abrasion test has now been recommended for acceptance by the British Standards Institution as a standard drum test and is specified as follows :

1. *Sample.*—The sample shall consist of about 100 lb. of coke sized by hand to pass a 3-in. and to remain on a 2-in. Coarse Mesh Test Sieve. The sample shall be prepared from a gross sample collected by increments distributed evenly over the whole consignment, and the pieces taken shall be representative of the sizes of the lumps over 2-in. in size in the consignment. The lumps over 3-in. in size shall be broken to pass a 3-in. and remain on a 2-in. B.S. Coarse Mesh Test sieve. The sample shall be dried.

2. *Apparatus.*—The drum abrasion test apparatus shall consist of a welded steel † drum of 30-in. internal diameter and 18-in. internal length and of not less than ¼-in. plate with a horizontal shaft of 1½-in. diameter passing through the drum and mounted on suitable bearings.

Two angles 2½ by 2½ by ⅜-in. shall be welded to the internal surface of the cylindrical portions of the drum at opposite ends of a diameter, and inclined at an angle of 17 degrees to a horizontal plane passing through the axis of the drum and the centres of the angles, the legs of which shall point in the direction of rotation of the drum. The two angles are mutually inclined (Fig. 27). The charging hole cover shall be fitted at 90 degrees to the horizontal plane passing through the axis of the drum and the centres of the angles and shall be flush with the internal surface of the

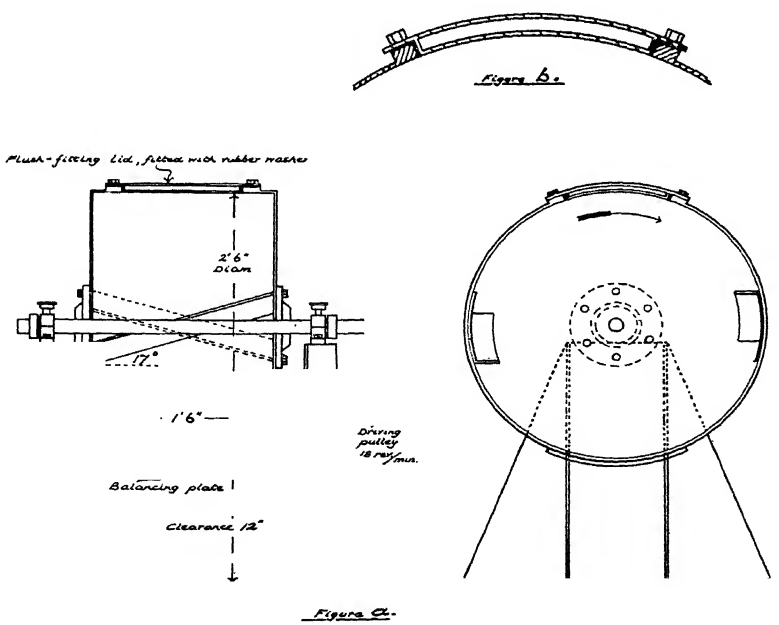


Fig. 27.—The Drum Abrasion Test Apparatus. (*a*) Vertical Sections through Drum ; (*b*) Charging -hole Cover.

drum, a convenient way of fitting, with an aperture 12 by 12-in. being illustrated in Fig. 27*b*. A balancing plate is welded to the outside of the drum, diametrically opposed to the charging hole cover. The bearings shall be mounted on suitable side supports to allow a clearance of at least 12-in. below the drum for convenient emptying after a test. The drum shall be driven at a speed of 18 ± 2 r.p.m. by suitable pulleys or gearing, a revolution counter being conveniently used to record the total number of revolutions. A rubber washer on the charging hole cover may be used to make it dust tight.

3. *Description of Test.*—Twenty-eight pounds of coke, dried or air-dry, screened by hand to pass a 3-in. and remain on a 2-in. B.S. Coarse Mesh Test sieve, shall be placed in the drum, the lid screwed on, and the drum rotated for 1,000 revolutions. The contents of the drum shall be emptied completely into a suitable container and shaken on a ⅛-in. B.S. Coarse Mesh Test sieve. The residue on the sieve shall be weighed to the nearest ounce and calculated as a percentage (given to one decimal place) of the original coke.

4. *Statement of Results.*—At least three tests shall be made, the results averaged and the mean shall be reported to the nearest whole number as the "B.S. Abrasion Index."

* The Drum Abrasion Test is based on that introduced by Mr. G. D. Cochrane in 1909, at Ormesby Ironworks, Co. Durham, as a modification of the drum test used by Sir I. Lowthian Bell (*J. Iron and Steel, Inst.*, 1904, **1**, 188). Cochrane, as a result of nine years experience of the test, stated that where the abrasion index fell below 74 the driving of the blast furnace deteriorated (*J. Iron and Steel Inst.*, 1918, **1**, 141), a result which has been confirmed by extensive use of the test at other blast-furnace plants (see, for example, *J. Iron and Steel Inst.*, 1936, **2**, 287P).

† Manganese steel resists wear better than mild steel.

CHAPTER X

THE HARDNESS OF COKE

THE SHATTER TEST

THE "impact hardness" or, simply, the "hardness" of coke is its power of resistance to breakage by impact, *e.g.* on being dropped into wagons or bunkers or to the charge-level of a blast-furnace.

The American standard "shatter test," for measuring the impact hardness of coke, was fully described in our earlier report. It has now been adopted as a British standard (*B.S. Specification for the Sampling and Analysis of Coke, No.* 496 of 1933), and is extensively used. The description of the apparatus remains as given in "Coke for Blast Furnaces" (p. 94), but the method of carrying out the test has been altered somewhat and is specified as follows:

"*Sampling for Shatter Test.* The sample shall be taken from the wagons leaving the maker's works or on arrival at the user's works.*

"The gross weight of the sample shall be 250-lb. collected by increments distributed evenly over the whole number of wagons in the consignment.

"Each increment shall consist of a single piece of coke over 2-in. in size, and the pieces taken shall be representative of the sizes of the lumps in the consignment.

"*Description of Test.* Fifty pounds of the coke screened by hand over a 2-in. British Standard Coarse Mesh Test Sieve, in such a way that any piece which can be passed through the screen in any position shall be counted as undersize, shall be placed in the box of the shatter test apparatus. The coke shall be levelled, the box raised, and the coke dropped on to the plate. The box shall be lowered and all the coke shovelled into it indiscriminately, but taking care to avoid breakage. The box shall then be raised and the coke again dropped. When the coke has thus been dropped four times in all, it shall be screened by hand over 2-in., 1½-in., 1-in., and ½-in. British Standard Coarse Mesh Test Sieves † as described above, and the weights of each grade shall be determined to the nearest ounce.

"*Statement of Results.* At least three tests shall be made and the results recorded as percentages (given to one decimal place) remaining on the screens.

"*Shatter Index.* The shatter index shall be the average of the percentages on the representative screens. In general, the shatter indices reported shall be:

1. The 2 -in. index.
2. The $1\frac{1}{2}$-in. index.

The shatter indices shall be given to the nearest whole number, except for the $\frac{1}{2}$-in. index which, when reported, shall be given to the first decimal place.

"*Tolerated Deviation.* If the average deviation of the individual results from their mean exceeds 2·5 and 1·5 units with the 2-in. and $1\frac{1}{2}$-in. screens respectively, a further series of at least two tests shall be made and the average of all the results taken."‡

B.S. Mesh Size. Nominal Size of Aperture (Side of Square)		Nominal Width of Bridge		Approximate Screening Area (per cent.)	Tolerance on Average Aperture, plus or minus per cent.
in.	mm.	in.	mm.		
$\frac{1}{2}$	12·7	0·192	4·88	52	2
1	25·4	0·300	7·62	59	2
$1\frac{1}{2}$	38·1	0·372	9·45	64	2
2	50·8	0·500	12·70	64	2

* The object of this proviso is to ensure that coke tested by both the maker and the user shall have received the same (a minimum) amount of dropping during handling.

† British Standard perforated plate for Coarse Mesh Test Sieves.

‡ The results are usually reported in the form 74 86, such figures representing the 2-in. and $1\frac{1}{2}$-in. indices.

It has been found that, in practice, screens of insufficient area to support the whole of the 50 lb. of coke taken have been used. Many of the screens, also, have been made with too wide a bridge and have in consequence been so heavy as to make the test difficult to carry out. The screens should be 2-ft. square and the width of bridge recommended in the British Standard should not be exceeded. The thickness of the plates should be $\frac{3}{16}$-in. for the $1\frac{1}{2}$-in. and larger, and $\frac{1}{8}$-in. for the 1-in. and smaller screens. The holes should be staggered.

The containers used for weighing the coke are often too heavy. Boxes made of $\frac{1}{16}$-in. sheet iron, 18 by 14 in. at the top and 14 by 10 in. at the bottom, 10 in. deep, conveniently hold 50 lb. of coke. Handles should be fixed on the short sides. Smaller containers 18 by 12 in. at the top and 14 by 12 in. at the bottom, 6 in. deep, are suitable for weighing the coke under 2 in. formed by the test. An Avery No. 118 counter platform weigher, of capacity 1 cwt. by 1 oz., is a satisfactory machine for the weighings.

Care must be taken to avoid loss of coke during a test; if necessary, the back and sides of the base of the shatter test apparatus should be boarded round. The whole of each of the different

sizes of coke formed by a test should be weighed as a check against loss. If the total weight is less than 50 lb. by more than 3 or 4 ounces the test should be suspect; it should not be assumed that the loss is due to "dust," for the weighings of the different fractions may each be slightly wrong. When converting the weights of the fractions, in ounces, into percentages, a rough adjustment for loss can be made, as in the following example:

Size	lb.	oz.	Per cent.	Per cent. adjusted
Over 2-in. .	33	11	67·375	67·5
Over 1½-in. .	6	3	12·375	12·4
Over 1-in. .	5	13	11·625	11·7
Over ½-in. .	3	1	6·125	6·2
thro' ½-in.	1	1	2·125	2·2
	49	13	99·625	100·0

In this adjustment, each of the calculated percentages is increased to the nearest 0·1 and an addition of 0·1 is made to the highest value (*i.e.*, the percentage over 2-in.). A conversion table for ounces to percentage of 50 lb. of coke, which can readily be memorised, is as follows:

Ounces	2	4	6	8	10	12	14
Percentages	0·12	0·25	0·37	0·50	0·62	0·75	0·87

Choice of the higher or the lower equivalent 0·1 percentage, *e.g.* 0·3 or 0·2 per cent., as equivalent to 4 ounces, is made according to whether the total weight of coke accounted for in the test is below (as is usual) or above 50 lb. When importance is to be attached to the ½-in. index, it is particularly desirable to follow this procedure and not to allow all "error" to accumulate on the "through ½-in." percentage.

The Accuracy of the Shatter Index

It is important to be able to judge of the degree of accuracy that can be assumed for a determined value of a shatter index. At many blast-furnace plants, it may only be practicable to make, perhaps, one shatter test on one wagon of a particular coke because so many different cokes are used. How far can the result of this one test be taken to indicate the average hardness of the coke in that wagon? At other plants, a shatter test may be made on a daily sample of coke made at ovens serving the blast-furnaces or a sample representative of the coke made during each shift may be tested. Can such a test truly assess the average value of the coke made per day or per shift?

The answers to these questions depend partly on the

"variability" of the coke and partly on the efficiency of the sampling. In blast-furnace practice some hundreds of tons of one make of coke may be used daily. Variations in the quality of the coke occur according to whether it comes from the top or the bottom of the ovens; or if the walls of some of the ovens in the battery are unevenly heated; or if in some of the ovens the walls are not quite straight and over-carbonisation is necessary to ensure that the coke shall be discharged satisfactorily; or if there are wide variations in the proportions of several coals that make up the oven charges.

The only way to discount the variability in hardness of a coke due to such causes is to make a number of shatter tests, the minimum number required for a specified "accuracy" depending on the degree of variability. The "variability" can be judged either by the average deviation of a large number of tests from the mean (sometimes termed the "average error"), or by some other factor calculated therefrom. Since knowledge of the "average error," of the "probable error" and of the chances that a specified "accuracy" can be obtained is of considerable importance, methods of calculating these data must be considered.

The True Shatter Index.—The true shatter index of a coke is the arithmetic mean of a large number, say 50, of tests the sampling for which has been carried out in such a manner that due allowance for variability in the quality of the coke has been made. For example, variation in the quality of the coke from an individual oven can be covered by taking a sufficiency of samples from different positions in the oven, or, in practice, from a belt conveyor taking the coke to the screening plant.

The Average Error.—The average error, or deviation from the mean value, is the sum of the arithmetic deviations of individual tests from the arithmetic mean divided by the number of tests. The average errors for the shatter indices of a Yorkshire coke on which 45 tests were made are recorded in Table XLVIII together with the results of the individual tests.

The coke was collected from one oven only and was made during a holiday period when a considerable proportion of "foreign" coking slack of inferior quality had to be used without adequate arrangements for blending it. The oven was of 20½-in. mean width, and the coal was top-charged and coked in 30 hours. About 36 cwt. of coke pieces were collected (coke from near the doors of the oven being excluded) and divided into six heaps. Size analyses of these heaps were made to determine whether the coke had been properly proportioned amongst them. The percentages of over 2-in. size in the heaps were 65, 62, 71, 65, 63 and 63, showing satisfactory distribution of the coke in all except heap No. 3. Each 50-lb. sample taken for shatter test was made up of over 4-in., 3-in., and

2-in. sizes in about the average proportion indicated by the size analyses of the six heaps. The tests are recorded in Table XLVIII fortuitously, not in the order in which they were made.

TABLE XLVIII.—AVERAGE ERRORS OF SHATTER INDICES, YORKSHIRE COKE.

Test No.	2-in.	Error	1½-in.	Error	1-in.	Error	½-in.	Error
6	68·1	1·4	79·3	1·6	88·2	−0·2	96·1	−0·1
25	68·3	1·6	78·9	1·2	88·4	0·0	96·2	0·0
10	68·9	2·2	78·4	0·7	90·1	1·7	96·4	0·2
20	63·2	−3·5	74·8	−2·9	88·7	0·3	96·0	−0·2
36	66·5	−0·2	75·8	−1·9	87·7	−0·7	96·2	0·0
11	69·1	2·4	78·7	1·0	88·6	0·2	96·5	0·3
8	65·6	−1·1	78·6	0·9	89·0	0·6	96·7	0·5
31	63·6	−3·1	75·2	−2·5	87·2	−1·2	96·0	−0·2
37	66·1	−0·6	76·2	−1·5	87·6	−0·8	95·6	−0·6
23	62·6	−4·1	78·1	0·4	88·8	0·4	96·8	0·6
39	64·7	−2·0	77·7	0·0	88·4	0·0	96·5	0·3
34	69·3	2·6	81·3	3·6	89·9	1·5	96·2	0·0
21	66·7	0·0	77·2	−0·5	88·3	−0·1	96·3	0·1
24	63·5	−3·2	79·0	1·3	89·1	0·7	96·7	0·5
5	69·1	2·4	81·0	3·3	90·1	1·7	97·2	1·0
14	67·0	0·3	77·1	−0·6	89·6	1·2	96·5	0·3
2	66·1	−0·6	78·3	0·6	88·9	0·5	96·6	0·4
43	66·6	−0·1	76·4	−1·3	87·4	−1·0	96·1	−0·1
18	64·5	−2·2	77·8	0·1	88·0	−0·4	96·0	−0·2
30	65·5	−1·2	75·0	−2·7	86·8	−1·6	95·7	−0·5
29	68·5	1·8	78·3	0·6	99·2	−0·2	96·0	−0·2
28	65·7	−1·0	76·8	−0·9	86·7	−1·7	95·7	−0·5
33	65·4	−1·3	75·8	−1·9	88·9	0·5	95·9	−0·3
7	66·3	−0·4	77·0	−0·7	88·5	0·1	96·4	0·2
38	64·9	−1·8	74·9	−2·8	86·5	−1·9	95·5	−0·7
19	66·9	0·2	78·4	0·7	89·5	1·1	96·2	0·0
17	67·5	0·8	79·2	1·5	88·4	0·0	96·5	0·3
16	68·0	1·3	79·8	2·1	88·9	0·5	96·1	−0·1
45	67·0	0·3	76·8	−0·9	86·9	−1·5	95·9	−0·3
35	70·3	3·6	78·8	1·1	88·7	0·3	96·1	0·1
9	69·6	2·9	78·2	0·5	89·9	1·5	96·6	0·4
3	70·6	3·9	80·0	2·3	89·5	1·1	96·4	0·2
44	65·3	1·4	75·8	−1·9	88·4	0·0	96·3	0·1
42	68·4	1·7	76·5	−1·2	88·5	0·1	96·9	0·7
41	67·8	1·1	76·8	−0·9	88·6	0·2	95·9	−0·3
13	63·1	−3·6	74·1	−3·6	86·2	−2·2	96·2	0·0
22	64·4	−2·3	78·0	0·3	88·1	−0·3	96·1	−0·1
27	68·0	1·3	78·0	1·1	88·9	0·5	96·4	0·2
15	64·9	−1·8	77·0	−0·7	88·4	0·0	95·9	−0·3
12	71·5	4·8	79·9	2·2	88·9	0·5	96·4	0·2
1	70·0	3·3	79·4	1·7	90·2	1·8	96·8	0·6
40	66·7	0·0	75·2	−2·5	87·0	−1·4	96·0	−0·2
4	65·9	−0·8	77·8	0·1	88·5	0·1	96·6	0·4
32	62·6	−4·1	77·4	−0·3	88·3	−0·1	96·0	−0·2
26	65·5	−1·2	79·3	1·6	88·4	0·0	96·2	0·0
Av.	66·7	1·81*	77·7	1·39*	88·4	0·72*	96·2	0·28*
Max.	71·5	4·8	81·3	3·6	90·2	2·2	97·2	1·0
Min.	62·6	0·0	74·1	0·1	86·2	0·0	95·5	0·0

* Arithmetic, not algebraic

These show that, for the coke from one oven, the 2-in., 1½-in., 1-in. and ½-in. shatter indices might be within ranges of 8·9, 7·2, 4·0 and 1·7. These ranges may be misleading, however, and it is more important to consider the average deviations from the mean values, the average deviation or "average error" being the best criterion of variability. For the 2-in. index the average deviation is 1·81 whilst for the ½-in. index it is 0·28.

The average error can be reduced by taking a group of tests as a unit. In Table XLIX the average errors, when from 2 to 20 of the tests in Table XLVIII are grouped, are compared with those calculated from the "probable errors," as explained later.

TABLE XLIX.—THE AVERAGE ERRORS OF GROUPS OF SHATTER TESTS.

No. of Tests Grouped	2-in. Index		1½-in. Index		1-in. Index		½-in. Index	
	Determined	Calculated	Determined	Calculated	Determined	Calculated	Determined	Calculated
1	1·81	—	1·39	—	0·72	—	0·28	—
2	1·32	1·28	0·95	0·98	0·50	0·51	0·19	0·20
3	1·08	1·04	0·85	0·80	0·41	0·42	0·17	0·16
4	0·67	0·90	0·72	0·69	0·36	0·36	0·13	0·14
5	0·70	0·81	0·59	0·62	0·36	0·32	0·14	0·12
6	0·76	0·74	0·44	0·57	0·26	0·29	0·15	0·11
8	0·52	0·64	0·58	0·49	0·28	0·25	0·11	0·10
9	0·45	0·60	0·49	0·46	0·23	0·24	0·13	0·09
10	0·48	0·57	0·23	0·44	0·14	0·23	0·10	0·09
12	0·51	0·52	0·16	0·40	0·09	0·21	0·02	0·08
15	0·21	0·47	0·23	0·36	0·23	0·19	0·11	0·07
20	0·48	0·40	0·10	0·31	0·08	0·16	0·09	0·06

In general, agreement between the determined and calculated values is good.

The Probable Error.—Although the "average error" is the best measure of the variability of a coke, it gives no indication of the number of results in a series which are likely to fall within a given tolerance of the true result. The "probable error," is that which 50 per cent. of the results do not exceed. In other words, amongst a large number of results, the chances that the error of any single test will not exceed the probable error are about even.

The probable error is related to the average error by Peter's approximation formula:

$$r = 0{\cdot}85\ a$$

in which r is the probable error and a is the average error. The approximation is only satisfactory when the number of tests is fairly large.*

* See "Report on the Sampling of Small Fuel up to 3-in., Embodying some General Principles of Sampling," *B.S.* No. 403, of 1930; and "The Experimental Error of the Shatter Test," by Briscoe and Marson, *Fuel in Science and Practice*,

The average and probable errors, for the $1\frac{1}{2}$-in. shatter index, of a number of cokes, with each of which about 50 tests were made, are recorded in Table L, together with the average indices and the ranges of the values obtained. For the tests on the cokes Yorks. I II and III, the product of one oven was used, and those on Yorks. IV were made over a period of about 3 weeks. The tests on the Scottish coke were made by the Scottish Coke Research Committee, and those on the Durham and Cumberland cokes by the Northern Coke Research Committee.

TABLE L.—AVERAGE AND PROBABLE ERRORS OF $1\frac{1}{2}$-IN. SHATTER INDICES OF COKES.

	D.I	D.II	Y.II	Y.III	S.I	Y.I	C.I	Y.IV
Mean shatter index	92·9	91·8	84·4	83·5	78·3	77·7	77·3	74·8
Maximum	95·6	94·2	88·0	87·4	82·6	81·3	81·5	79·1
Minimum	90·1	90·0	81·2	80·8	73·5	74·1	72·5	70·8
Average error	0·93	0·68	1·25	1·07	1·59	1·39	1·52	0·85
Probable error	0·79	0·58	1·06	0·91	1·35	1·18	1·29	0·72

D. = Durham. Y. = Yorks. S. = Scottish. C. = Cumberland.

These probable errors relate to single tests. Taking coke Durham I as an example, the meaning of the value for the probable error is that the $1\frac{1}{2}$-in. shatter index of that coke as determined in a single shatter test is just as likely as not to be within the range of 92·9 ± 0·79, *i.e.* within the range of 93·7 to 92·1.

If greater accuracy than this is required for the shatter index the results of a number of tests must be averaged. The probable error is then $\frac{\text{---}}{\sqrt{n}}$, n being the number of tests. Thus, for coke Durham I, the probable errors if the results of groups of 2, 3 or 5 tests were averaged would be 0·56, 0·46 or 0·35, and there would be an even chance of the value of the $1\frac{1}{2}$-in. index lying within the ranges 93·5–92·3, 93·4–92·4 or 93·3–92·5, when the index is obtained by averaging 2, 3 or 5 tests.

It is evident that the probable error will be less the larger the number of tests averaged. Hence the recommendation in the B.S. specification that, when reporting a shatter index, the results shall be the mean of 3 or 5 tests according to the average deviation. For example, if the average deviation ("error") of the $1\frac{1}{2}$-in. index is less than 1·5, three tests suffice; if it is greater than 1·5, five tests should be made and the results averaged. With an average error of 1·3, with a group of 3 tests, the probable error would be 0·64;

1931, **10**, 466. The simple relationship $r = 0{\cdot}85\ a$, is, for our present purpose, as satisfactory as the formula $r = \frac{0{\cdot}8453\ a}{\sqrt{n\,(n-1)}}$ used by Briscoe and Marson (a is the sum of the average errors and n is the number of tests).

and with an average error of 1·8, with a group of 5 tests, the probable error would be 0·7. For general purposes, therefore, the shatter test as ordinarily carried out, with the indices recorded as the nearest whole numbers, gives sufficiently accurate results. For tests of cokes made in our experimental oven we have usually made more than three shatter tests if the average error of the $1\frac{1}{2}$-in. index has exceeded 1·0; the probable error of the mean result is then less than 0·5 and the results may be recorded to the nearest decimal place.

Errors Having Greater than Even Chance.—When a high degree of accuracy is required in reporting the shatter index, it is necessary to consider errors having greater than an even chance of occurring. For example, it may be necessary to specify that an error of 0·5 shall not be exceeded in 10 per cent. of reports made; or that such an error shall not occur more than once in 100 reports.

For such purposes the chances of the specified error occurring can be calculated by the probability integral:

$$P = \frac{2}{\sqrt{\pi}} \int_0^{\frac{kx}{r}} e^{-\left(\frac{x}{r}\right)^2} d\left(\frac{x}{r}\right) \quad . \quad . \quad . \quad . \quad (1)$$

in which P is the probability that an error x will occur when the probable error is r, k being a constant $=0{\cdot}477$. Values for the probability integral:

$$P = \frac{2}{\sqrt{\pi}} \int_0^{y} e^{-t^2} \, dt \quad . \quad . \quad . \quad . \quad . \quad (2)$$

in which $y = \frac{kx}{r}$ and $t = \frac{x}{r}$, are given in mathematical tables, and are reproduced in the appendix to this chapter in terms of y and P.

For example, for coke Yorks III (Table L) $r=0{\cdot}91$, and for different values of error x the term $y = \frac{kx}{r}$ in the expression (2) can be calculated. Thence, from the table given in the appendix to this chapter, values of P for different values of y can be obtained as follows:

$x = 0{\cdot}5$,	$y = 0{\cdot}262$	$P = 0{\cdot}289$,	= odds of 0·4 : 1
$x = 1{\cdot}0$,	$y = 0{\cdot}524$	$P = 0{\cdot}542$,	= odds of 1·2 : 1
$x = 2{\cdot}0$,	$y = 1{\cdot}048$	$P = 0{\cdot}862$,	= odds of 6·2 : 1

The odds are thus 6·2 to 1 that an error of $\pm 2{\cdot}0$ will not be exceeded, in a single test, in the value for the $1\frac{1}{2}$-in. shatter index of coke Yorks. III.

Probabilities can similarly be worked out for different errors and groups of any number of tests averaged for a shatter index, using the appropriate probable errors. Fig. 28 shows in graphical

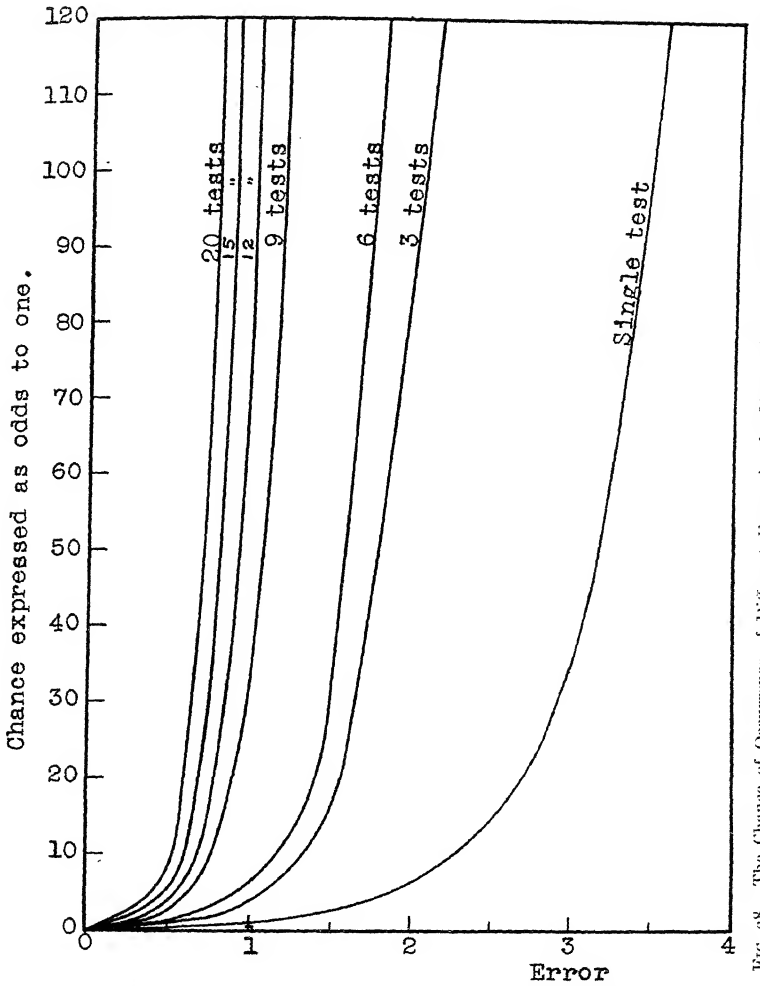


FIG. 28.—The Chance of Occurrence of Different Errors in the Mean $1\frac{1}{2}$-in. Shatter Index. Yorks. III Coke with Various Groups of Tests Averaged.

form the chances of different errors occurring in the $1\frac{1}{2}$-in. shatter index of coke Yorks. III for single tests and for groups of 3 to 20 tests. The rapid fall in magnitude of the error that can occur once in 100 times, or even once in 10 times, if, instead of taking the result of a single test, three tests are grouped, should be noted. From such graphs for a number of different cokes, the errors with 9 to 1 and 99 to 1 probabilities for single tests and for various groups of tests have been determined and are recorded in Tables LI and LII.

TABLE LI.—ERRORS OF 9 TO 1 PROBABILITY IN $1\frac{1}{2}$-IN. SHATTER INDEX. SINGLE TESTS AND GROUPS OF TESTS.

Coke	Shatter Index ($1\frac{1}{2}$-in.)	Errors—No. of Tests Grouped						
		Single Test	3	6	9	12	15	20
Durham I . .	92·9	2·0	1·2	0·8	0·7	0·6	0·5	0·4
Durham II . .	91·8	1·5	0·8	0·6	0·5	0·4	0·4	0·3
Yorks. II . .	84·4	2·7	1·6	1·1	0·9	0·8	0·7	0·6
Yorks. III . .	83·5	2·3	1·3	0·9	0·8	0·7	0·6	0·5
Scot. I . . .	78·3	3·4	2·0	1·4	1·1	1·0	0·9	0·8
Yorks. I . .	77·7	3·0	1·7	1·2	1·0	0·9	0·8	0·7
Cumb. I . .	77·3	3·3	1·9	1·3	1·1	1·0	0·8	0·7
Yorks. IV . .	74·8	1·7	1·0	0·7	0·6	0·5	0·5	0·4

TABLE LII.—ERRORS OF 99 TO 1 PROBABILITY IN $1\frac{1}{2}$-IN. SHATTER INDEX. SINGLE TESTS AND GROUPS OF TESTS.

Coke	Shatter Index ($1\frac{1}{2}$-in.)	Errors—No. of Tests Grouped						
		Single Test	3	6	9	12	15	20
Durham I . .	92·9	3·1	1·8	1·2	1·0	1·9	0·8	0·7
Durham II . .	91·8	2·2	1·3	0·9	0·7	0·6	0·6	0·5
Yorks. II . .	84·4	4·0	2·3	1·7	1·4	1·1	1·0	0·9
Yorks. III . .	83·5	3·5	2·0	1·5	1·2	1·0	0·8	0·8
Scot. I . . .	78·3	5·3	3·0	2·1	1·7	1·5	1·4	1·2
Yorks. I . .	77·7	4·6	2·6	1·9	1·5	1·3	1·1	1·0
Cumb. I . .	77·3	5·0	2·9	2·0	1·7	1·4	1·3	1·1
Yorks. I . .	74·8	2·8	1·5	1·1	0·9	0·8	0·7	0·6

As examples of the use of the values in Tables LI and LII, it will be seen that for coke Durham II, if a single shatter test is made, any error will not exceed $\pm 1\cdot5$ in 9 out of 10 occasions (or in 90 out of 100), whilst for the Scottish coke an error of $\pm 3\cdot4$ will not be exceeded. If the shatter index taken is the average of 3 tests, the error will not exceed $\pm$ 2·0 for any of the cokes, in 90 out of 100 occasions, and it will not exceed $\pm$ 3·0 in 99 out of 100 occasions.

The Number of Shatter Tests Required to Ensure a Specified Degree of Accuracy.—From Fig. 28 one can determine for Yorks. III coke the errors that have a given chance of occurring, say a 9 to 1 or a 99 to 1 chance, dependent on the number of tests grouped to provide a $1\frac{1}{2}$-in. shatter index. The nests of curves drawn in Figs. 29 and 30 have been constructed for different values of the probable error, r, of groups of tests as given by formula (2), the data being :

Probability 9 : 1. $P = \frac{9}{10} = 0{\cdot}9000$. $y = 1{\cdot}163^* = \frac{0{\cdot}477x}{r}$, or $x = 2{\cdot}44r$.

Probability 99 : 1. $P = \frac{99}{100} = 0{\cdot}9900$. $y = 1{\cdot}82^* = \frac{0{\cdot}477x}{r}$, or $x = 3{\cdot}82r$

From Figs. 29 and 30 the values in Table LIII have been taken.

TABLE LIII.—THE NUMBER OF SHATTER TESTS REQUIRED TO ENSURE A SPECIFIED DEGREE OF ACCURACY.

Coke	Mean Shatter Index	Average Error	Probable Error	No. of Tests Required: for Error ± 1·0		for Error ± 0·5	
				Probability		Probability	
				9 : 1	99 : 1	9 : 1	99 : 1
Durham I .	92·9	0·93	0·79	4	9	16	36
Durham II .	91·8	0·68	0·58	2	5	8	20
Yorks. II .	84·4	1·25	1·06	7	16	26	66
Yorks. III .	83·5	1·07	0·91	5	12	22	48
Scot. I . .	78·3	1·59	1·35	12	28	43	107
Yorks. I . .	77·7	1·39	1·18	9	20	34	81
Cumb. I . .	77·3	1·52	1·29	10	25	41	97
Yorks. IV .	74·8	0·85	0·72	3	8	14	31

If the average error does not exceed 1·5, the shatter index can be determined within ±0·5 of the true value on 9 occasions out of 10 by averaging the results of not more than 38 tests. If, however, it is desired that an error of ±0·5 shall not be exceeded once in 100 times, an average of 38 tests would only give a satisfactory result if the average error were less than 0·9; if the average error were 1·5, more than 100 tests would have to be averaged.

The theory of errors enables calculation to be made of the number of results of a series of tests which would lie within a specified range of error. For example, supposing that with 99 results out of 100 the error does not exceed ±1·0, it can be shown that 50 of these results would lie within ±0·26 of the correct result, and that 80 would lie within ±0·5†. It is evident that such a high standard of accuracy may not be necessary, and that the practical difficulty of making a large number of tests, when the average error of the coke is as high as 1·5, can be met by adopting a lower standard. An error of ±1·0 with a probability of 9 to 1 would meet most practical requirements. The number of tests to be averaged would then not exceed 10 if the average error of the coke did not exceed 1·5.

* The values of y are found from the table in the appendix for the appropriate values of P.

† For a probability 0·9900 (i.e. 99 out of 100), the value of y in the probability integral (see Appendix, p. 131) is 1·82. We are considering a case in which the error x does not exceed ± 1·0 in 99 cases out of 100. Hence, since, $y = 1{\cdot}82 = \frac{0{\cdot}477\,x}{r}$

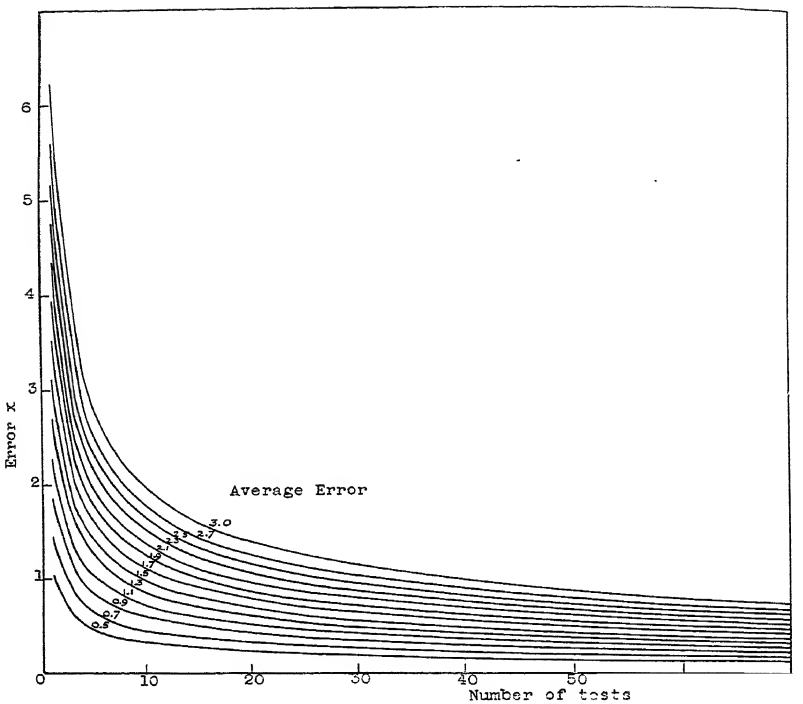


FIG. 29.—Probability 9 : 1. Errors which may occur once in 10 times in the Averages of Various Groups of Tests. Cokes of different Average Error in $1\frac{1}{2}$-in. Shatter Index.

and $x = 1$, r the probable error for this particular case $\frac{0 \cdot 477}{1 \cdot 82}$ 0·26. Now, considering an error $x = 0 \cdot 26$, $y = \frac{0 \cdot 477 \times x}{r}$ and $x = 0 \cdot 26 = r$, so that $y = 0 \cdot 477$. For this value of y, P = 0·50. In other words, the error x does not exceed $\pm$ 0·26 in 50 per cent. of the cases, if the error is not greater than $\pm$ 1·0 in 99 of 100 cases.

For other values of the error x the following values are calculated :

x	y	P	Percentage of cases with error within $\pm x$
0·1	0·182	0·20	20
0·2	0·364	0·39	39
0·3	0·545	0·56	56
0·4	0·726	0·69	69
0·5	0·908	0·80	80
0·6	1·090	0·88	88

Similarly, when 9 out of 10 tests are correct to within $\pm$ 1·0, 7 are within $\pm$·60, 6 within $\pm$ 0·5, 4 within $\pm$ 0·4 and 3 within $\pm$ 0·3 of the correct result.

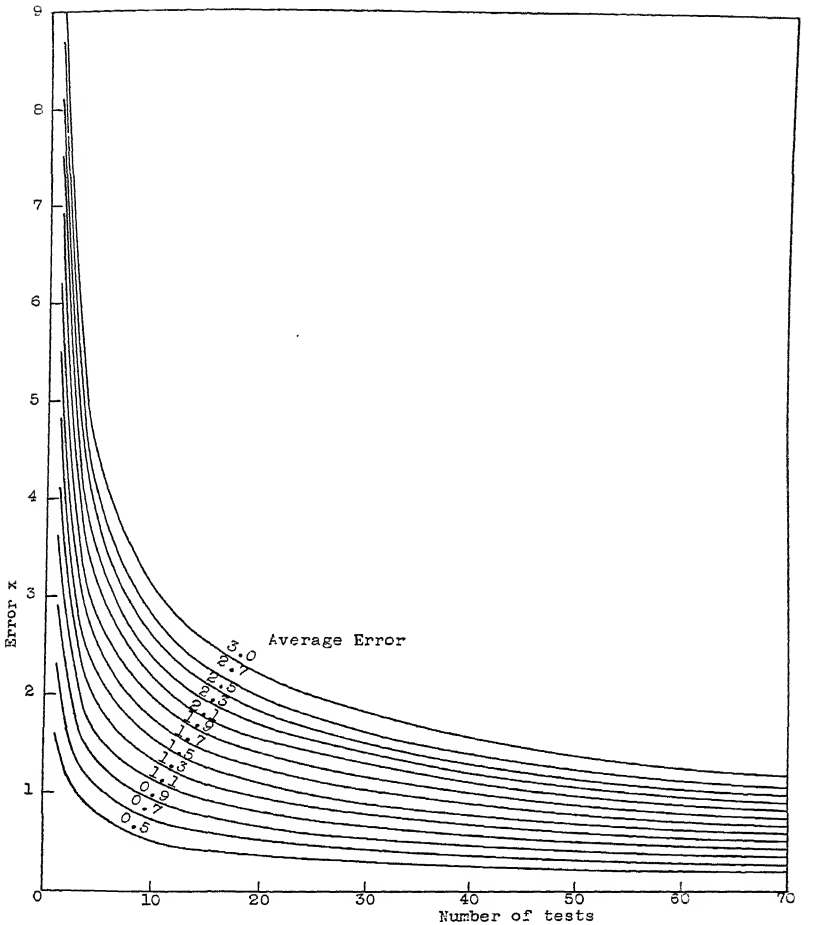


FIG. 30.—Probability 99 : 1. Errors which may occur once in 10 times in the Averages of Various Groups of Tests. Cokes of different Average Error in 1½-in. Shatter Index.

In Table LIV the average errors of the 1½-in. shatter index for a number of Midland cokes are recorded. These values are deduced from tests made at a blast-furnace plant, mostly over a period of 12 to 18 months. During such a long period all the factors upon which the variability of a coke depends must be presumed to operate on occasion, and the average errors can be regarded as extreme values.

TABLE LIV.—THE AVERAGE ERRORS FOR THE $1\frac{1}{2}$-IN. SHATTER INDEX. MIDLAND COKES.

Plant No.	Mean $1\frac{1}{2}$-in. Shatter Index	Average Error	No. of Tests	Period	Remarks
10	78·9	3·70	34	5 quarters	
4	72·0	3·53	29	5 ,,	Cake charge.
21	77·9	3·06	58	2 ,,	—
13	80·1	3·01	34	5 ,,	Cake charge.
6	75·2	2·89	34	5 ,,	Cake charge.
21	79·2	2·54	50	3 ,,	—
7	82·9	2·54	32	5 ,,	Cake charge.
20	79·2	2·41	15	5 ,,	Cake charge.
23	77·7	2·40	24	5 ,,	—
23	79·2	1·24	19	4 ,,	—
16	76·7	2·30	46	5 ,,	Modern ovens.
8	79·3	2·12	38	5 ,,	—
15	73·7	2·12	29	5 ,,	—
1	76·3	2·07	37	5 ,,	Cake charge.
9	75·2	2·06	31	5 ,,	Cake charge.
3	73·8	1·72	49	5 ,,	Cake charge.
14	79·5	1·69	17	4 ,,	Modern ovens.
5	83·2	1·44	31	5 ,,	—
11	84·8	1·44	26	5 ,,	—
21	77·7	1·39	50	1 day	—
21	84·4	1·25	50	1 ,,	—
22	87·9	1·25	50	2 quarters	—
22	88·1	1·15	52	2 ,,	—
2	80·5	1·09	18	5 ,,	—
14	83·5	1·08	50	1 day	Single seam.
25	77·9	1·02	24	5 quarters	Modern ovens.
17	79·4	1·02	44	5 ,,	Modern ovens.
12	88·5	0·98	21	4 ,,	Modern ovens.
46	74·8	0·85	50		Modern ovens.

With more than half these cokes the average error of the shatter index exceeded 1·5. It will be seen that, on the whole, the average error was least for the cokes made in modern ovens, not more than 18-in. wide and top-charged, whilst none of the cokes for which the average error was low was made from a cake charge. These are generalities, and it does not necessarily follow that coke made in modern, narrow ovens is more uniform in quality than that made in older ovens; coke No. 16 (average error 2·3), for example, was made in the same type of oven as coke No. 46 (average error 0·85).

The values for coke No. 23 are interesting. Over a period of 15 months the average error was 2·4, but the exclusion of low values for the shatter index obtained during one period of 3 months, when inferior coals were imperfectly blended, reduced the average error to well below 1·5 for the year.

The general conclusions to be drawn from this discussion of the degree of accuracy to be expected of the shatter index can be summarised thus :

(1) The accuracy with which the shatter index of a coke can be determined depends on the "variability" of the coke; this is indicated by the "average error" or deviation from the mean of a number of results of shatter tests.

(2) The "probable error," that is to say, the error which has an even chance of occurring, is 0·85 times the average error.

(3) If the probable error of one determination of the shatter index is r, it is $\frac{r}{\sqrt{n}}$ when the results of n tests are averaged; the greatest proportional decrease in the error is therefore made on increasing the number of tests from 1 to 3. Whenever possible, the shatter index should be the average of at least 3 tests.

(4) When a comparison of different cokes is required, it is desirable to limit the error in the determination of the shatter index to $\pm 1 \cdot 0$, and to assume the probability of such an error occurring to be not more than once in 10 determinations. For this degree of accuracy, each determination should usually be the average of 7 shatter tests, though the minimum number of tests that should be averaged depends upon the variability of the coke.

The Influence of the Hardness of Coke on Blast Furnace Performance

Members of the Midland Coke Research Committee have frequently given examples of the value of a hard coke in blast-furnace practice. For example, in Northamptonshire, it is a common practice to use a certain proportion of Welsh coke, the hardest made in this country, in admixture with Midland cokes, it having been found that the rate of drive of the furnace is improved thereby. Similarly, in Derbyshire, the admixture with Derbyshire cokes of the harder Yorkshire cokes is found to be advantageous.

A recent series of tests (Iron and Steel Institute, Blast-Furnace Committee Report No. 17) showed that, on replacing a mixture of two-thirds Yorkshire coke ($1\frac{1}{2}$-in. shatter index 77) and one-third Durham coke ($1\frac{1}{2}$-in. shatter index about 90) with a mixture containing, in successive weeks, 52 and 71 per cent. of a Continental coke ($1\frac{1}{2}$-in. shatter index 95), the rate of drive of the furnace increased from 1630 to 1838 and 2247 tons of pig-iron, the fuel-consumption being $21\frac{1}{2}$, 20 and 19 cwt. per ton of pig-iron.

The principal effect of the presence of a proportion of hard coke in the burden of a blast-furnace is to ensure a satisfactory rate of drive of the furnace. A similar effect has been obtained by screening out breeze, under $1\frac{1}{4}$-in., from a Yorkshire coke of less than average hardness (*see* p. 25), when an increase in the rate of drive of the furnace was accompanied by decreased coke-consumption. There is little doubt that the use of cokes harder than the average present qualities of Midland cokes would likewise be beneficial.

The Sampling of Coke for Shatter Test.—To be representative, a sample of coke for shatter test should include pieces from different ovens and from different positions in the ovens. Collection of a sample of this character could be made by hand as the coke moves along a conveyor, but in practice it is found more satisfactory (to avoid the unconscious selection of the better pieces) to take shovelfuls of coke from the conveyor at regular intervals. By such a method of sampling, all sizes of coke over 2-in. are represented.

If this method of sampling be adopted, each "increment" would weigh from 5 to 10 lb., and from 5 to 10 such increments would provide a sufficient quantity of coke (50 lb.) for one shatter test. A sample composed of 5 increments could hardly be representative of the output of one coke-making shift, and it is wiser to collect from 10 to 20 increments and thus obtain sufficient coke for 3 shatter tests.

APPENDIX

Solutions of the Probability Integral $P = \frac{2}{\sqrt{\pi}} \int^{y} e^{-t^2} dt$.

y	P	y	P	y		y	
·01	·0113	·70	·6778	1·39	·9507	2·08	·9967
·02	·0226	·71	·6847	1·40	·9523	2·09	·9969
·03	·0338	·72	·6914	1·41	·9539	2·10	·9970
·04	·0451	·73	·6981	1·42	·9554	2·11	·9971
·05	·0564	·74	·7047	1·43	·9569	2·12	·9973
·06	·0676	·75	·7112	1·44	·9583	2·13	·9974
·07	·0789	·76	·7175	1·45	·9597	2·14	·9975
·08	·0901	·77	·7238	1·46	·9610	2·15	·9976
·09	·1013	·78	·7300	1·47	·9624	2·16	·9978
·10	·1125	·79	·7361	1·48	·9637	2·17	·9979
·11	·1236	·80	·7421	1·49	·9649	2·18	·9980
·12	·1348	·81	·7480	1·50	·9661	2·19	·9981
·13	·1459	·82	·7538	1·51	·9673	2·20	·9981
·14	·1570	·83	·7595	1·52	·9684	2·21	·9982
·15	·1680	·84	·7651	1·53	·9695	2·22	·9983
·16	·1790	·85	·7707	1·54	·9706	2·23	·9984
·17	·1900	·86	·7761	1·55	·9716	2·24	·9985
·18	·2010	·87	·7814	1·56	·9726	2·25	·9985
·19	·2118	·88	·7867	1·57	·9736	2·26	·9986
·20	·2227	·89	·7918	1·58	·9746	2·27	·9987
·21	·2335	·90	·7969	1·59	·9755	2·28	
·22	·2443	·91	·8019	1·60	·9763	2·29	
·23	·2550	·92	·8068	1·61	·9772	2·30	·9989
·24	·2657	·93	·8116	1·62	·9780	2·31	·9989
·25	·2763	·94	·8163	1·63	·9788	2·32	·9990
·26	·2869	·95	·8209	1·64	·9796	2·33	·9990
·27	·2974	·96	·8254	1·65	·9804	2·34	·9991
·28	·3079	·97	·8299	1·66	·9811	2·35	·9991
·29	·3183	·98	·8342	1·67	·9818	2·36	·9992
·30	·3286	·99	·8385	1·68	·9825	2·37	·9992
·31	·3389	1·00	·8427	1·69	·9831	2·38	·9992
·32	·3491	1·01	·8468	1·70	·9838	2·39	·9993
·33	·3593	1·02	·8508	1·71	·9844	2·40	·9993
·34	·3694	1·03	·8548	1·72	·9850	2·41	·9994
·35	·3794	1·04	·8587	1·73	·9856	2·42	·9994
·36	·3893	1·05	·8624	1·74	·9861	2·43	·9994
·37	·3992	1·06	·8661	1·75	·9867	2·44	·9994
·38	·4090	1·07	·8698	1·76	·9872	2·45	·9995
·39	·4187	1·08	·8733	1·77	·9877	2·46	·9995
·40	·4284	1·09	·8768	1·78	·9882	2·47	·9995
·41	·4380	1·10	·8802	1·79	·9886	2·48	·9996
·42	·4475	1·11	·8835	1·80	·9891	2·49	·9996
·43	·4569	1·12	·8868	1·81	·9895	2·50	·9996
·44	·4662	1·13	·8900	1·82	·9899	2·51	·9996
·45	·4755	1·14	·8931	1·83	·9904	2·52	·9996
·46	·4847	1·15	·8961	1·84	·9907	2·53	·9997
·47	·4938	1·16	·8991	1·85	·9911	2·54	·9997
·48	·5028	1·17	·9020	1·86	·9915	2·55	·9997
·49	·5117	1·18	·9048	1·87	·9918	2·56	·9997
·50	·5205	1·19	·9076	1·88	·9922	2·57	·9997
·51	·5292	1·20	·9103	1·89	·9925	2·58	·9997
·52	·5379	1·21	·9130	1·90	·9928	2·59	
·53	·5465	1·22	·9155	1·91	·9931	2·60	·9998
·54	·5549	1·23	·9180	1·92	·9934	2·61	·9998
·55	·5633	1·24	·9205	1·93	·9937	2·62	·9998
·56	·5716	1·25	·9229	1·94	·9939	2·63	·9998
·57	·5798	1·26	·9252	1·95	·9942	2·64	·9998
·58	·5879	1·27	·9275	1·96	·9944	2·65	·9998
·59	·5959	1·28	·9297	1·97	·9947	2·66	·9998
·60	·6039	1·29	·9319	1·98	·9949	2·67	·9998
·61	·6117	1·30	·9340	1·99	·9951	2·68	·9999
·62	·6194	1·31	·9361	2·00	·9953	2·69	·9999
·63	·6271	1·32	·9381	2·01	·9955	2·70	·9999
·64	·6346	1·33	·9400	2·02	·9957	2·71	·9999
·65	·6420	1·34	·9419	2·03	·9959	2·72	·9999

CHAPTER XI

THE REACTIVITY OF COKE

THE word "reactivity" in relation to coke is somewhat loosely used to express the ability of a coke to interact with, usually, oxygen, carbon dioxide or steam. Frequently, no attempt is made to discriminate between the specific reactivity, an inherent quality of the coke substance (or, perhaps, the coke structure), and physical properties, such as size and density, which may affect the rate of combustion of a given kind of coke.

The specific reactivity of coke has been determined in several different ways; for example, by the so-called "critical air blast" or "C.A.B." (Blayden, Noble and Riley, *Journ. Inst. Fuel.*, 1934, **7**, 139), or by the rate of interaction of a powdered and carefully sized sample with either chromic acid (Blayden and Riley, *Journ. Soc. Chem. Ind.*, 1935, **54**, 159) or alkaline potassium permanganate, as used for coals by Francis (*Fuel in Science and Practice*, 1933, **12**, 128) and Heathcoat (*Fuel in Science and Practice*, 1933, **12**, 4).

The use of the C.A.B. and the chromic acid methods is referred to again later in this chapter. Less frequent use has been made of alkaline potassium permanganate for measuring the reactivity of coke. Some relative values, given by what are termed "permanganate numbers" (*i.e.* c.c. of N/10 potassium permanganate decomposed by 0·5 gramme of the dry, ash-free cokes at 100° C.), are recorded in Table LV together with corresponding values for different coals.

TABLE LV.—RELATIVE REACTIVITIES OF COKES AND COALS.

	(Permanganate Numbers).
Ceylon Graphite	7
Beehive coke from small coal	8
Parkgate hards coked in lumps in beehive oven	9
Hearth coke	21
Two Foot Nine coal (S. Wales), 89.8% C.* ..	23
No. 5 coal (Kent), 86.8% C.	36
Swallow Wood coal (S. Yorks.), 81.8% C. ..	56
Beeston coal (W. Yorks.), 81.1% C.	83
Main coal (Leicester.), 76.5% C.	89

* The percentages of carbon in the coals are on a dry, ash-free basis.

It will be seen that differentiation between high-temperature cokes by means of their "permanganate numbers" may not be easy, unless the method of test can be suitably modified. With low-temperature cokes, however, the values readily distinguished not only between cokes made from different coals but between cokes made from the same coal at different temperatures, as the records in Table LVI show.

TABLE LVI.—RELATIVE REACTIVITIES OF COKES MADE AT DIFFERENT TEMPERATURES (PERMANGANATE NUMBERS).

Coal Seam	Carbon, % dry, ash-free	Permanganate Number of Coal	Permanganate Numbers of Cokes made at :						
			300° C.	400° C.	500° C.	600° C.	700° C.	800° C.	900° C.
Ryder (S. Staffs.)	81·5	87	78	46	25	13	13	—	—
Barnsley (S. Yorks.)	84·4	29	29	36	22	10	9	—	—
Parkgate (S. Yorks.)	86·9	25	25	46	28	11	8	8	8
Two Foot Nine (S. Wales)	89·7	17	19	23	60	17	9	7	6

The interesting changes in the reactivities of cokes obtained within the temperature range of 300-500° C. are discussed later. It will be seen that, in general, the cokes made at temperatures of 700° C. or higher followed the same order of reactivity as the coals from which they were made.

Except when a coke is used in such a manner that a high combustion-temperature is not produced (*e.g.* in an open grate), it is doubtful whether measurement of its specific reactivity provides information of much value as to its probable behaviour. The conditions of burning of a high-temperature coke usually lead to high combustion-temperatures, when the most important factor affecting its general "reactivity" is its size.

THE INFLUENCE OF THE SIZE OF COKE

Under the head of "size" it is convenient to group a number of properties of coke all of which influence its rate of burning or its rate of reaction with carbon dioxide. In this respect, the surface-area is the most important of the attributes of size. The smaller the screen-size of pieces of coke the greater is the area of surface at which reaction can occur with a given volume of coke. The relationship between screen-size and surface-area is illustrated in Table LVII for one cu. ft. of a Yorkshire coke.

TABLE LVII.—SURFACE-AREAS OF ONE CU. FT. OF DIFFERENT SIZES OF COKE.

(Coke No. 31 C. Beehive Coke from Halifax Soft seam, S. Yorks.)

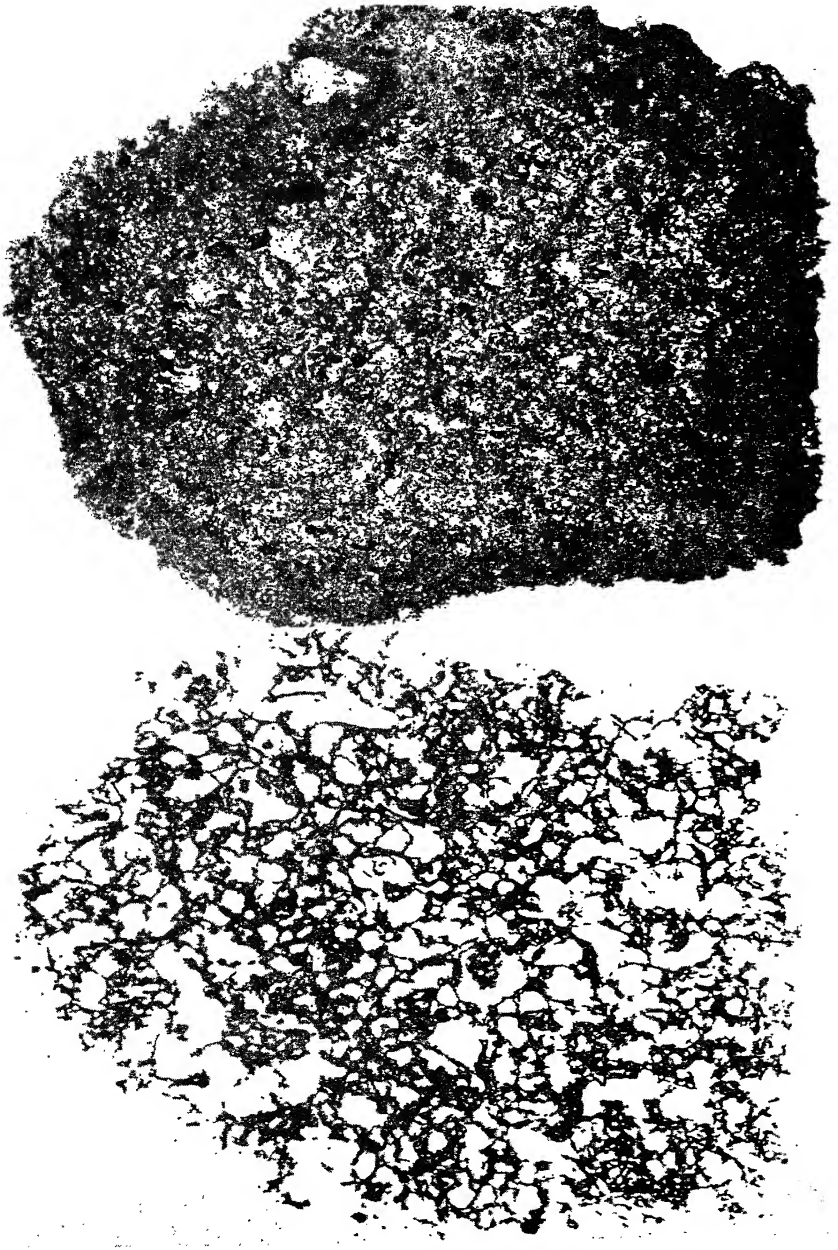
Size of Coke (sq. mesh. in.)	4–3½	3½–3	3–2½	2½–2	2–1½	1½–1	1–½
Apparent Sp. G. . .	0·88	=55 lb.	of solid	coke pe	r cu. ft.		
Bulk density (lb. per cu. ft.) . . .	26	26	26	24	29	29	31
Actual space occupied (cu. ft.) . . .	0·475	0·475	0·475	0·44	0·53	0·53	0·565
Surface area (sq. ft.) .	9·0	9·3	10·5	13·9	21·0	30·6	53·6
Free space in 1 cu. ft. (cu. ft.) . . .	0·525	0·525	0·525	0·56	0·47	0·47	0·435
Pore space in 1 cu. ft. (cu. ft.) . . .	0·25	0·25	0·25	0·23	0·27	0·27	0·29
Pore surface in 1 cu. ft. (sq. ft.) . . .	456	456	456	420	494	494	528

The assumption is made in the calculations in Table LVII that the pieces of coke are cubical, the sides of the cubes being those of the mean screen meshes. The total porosity is 51·8 per cent. of the apparent volume of the coke, and the pore-surface is calculated on the assumption that the mean diameter of the pores is 1 mm.

The bulk-density of a coke tends to increase as the size of the pieces decreases. With some cokes this increase is regular, with others there is a critical size at which the increase begins, and there may be discontinuity because a particular size may be difficult to pack owing, for example, to its shape being prismatic.

If the pore-surface of coke is effective surface for reaction with oxygen or carbon dioxide, the size of the pieces and their external surface-areas should be of comparatively small importance in influencing the rate of reaction; for as between 4–3-in. and 1–½-in. sizes the total surface (pore and external) of 1 cu. ft. of coke is no more than 25 per cent. greater for the smaller size (581 sq. ft. as compared with 465 sq. ft.). If, however, the external surface is of major importance, the size of the pieces must be a critical factor as regards the rate of combustion; for the surface-area of a given volume of coke of 1-½-in. size is six times as great as that of 4–3-in. size.

The pressure required to force air at the rate of 1 c.c. per minute through the pores of an average metallurgical coke is about 0·2 mm. W. G. for a cube of 1 cm. side. In a furnace 2 ft. in diameter in which a 2-ft. bed of coke was blown with air at a rate of about 90 cu. ft. per minute (to burn 1–½-in. size coke at a rate of 24 lb. per sq. ft. of grate area per hour), a pressure of 0·5-in. W.G. was required to overcome the resistance of the bed. This pressure, it can be calculated, would only pass through the pores of one piece of coke of 1–½-in. size a quantity of air equivalent to 0·4 per cent.



of the total supplied to it. When larger sizes of coke were used in the furnace, the pressure required to pass the same quantity of air through the bed was only 0·2-in. W.G., so that the amount of air which would pass through the pores of the pieces of coke would be correspondingly less.

With a blast-pressure of 7 lb. per sq. in., the amount of air that could pass through cubes of coke of 2-in. size can be calculated to be about 1 per cent. of the total blast supplied to burn coke at a rate of 100 lb. per sq. ft. of hearth-area per hour in a blast-furnace. It is evident, therefore, that the normal combustion of ordinary coke is mainly confined to the outer surface. This deduction has been confirmed by a study of the pore-structures of pieces of coke of different sizes, taken from different levels in a 2-ft. bed within a furnace 2 ft. in diameter, which was blown with sufficient air to give a rate of combustion of 20 lb. per sq. ft. of grate-area per hour. The density and porosity of the pieces of coke were determined, and samples were cut in two for the preparation of "Rose" sections with plaster-of-paris. Table LVIII records the mean results of the measurements of porosity.

TABLE LVIII.—POROSITY OF PIECES OF COKE AT DIFFERENT LEVELS OF A FURNACE (COKE No. 31C).

Size of Coke (in.)	4–3½		3½–3		3–2½		1½–1		1–½	
Distance above Grate (in.)	Total Pores %	Closed Pores %	Total Pores %	Closed Pores %	Total Pores %	Closed Pores %	Total Pores %	Closed Pores %	Total Pores %	Closed Pores %
18 – 13½	57·6	4·0	58·8	7·2	59·3	5·4	57·3	8·3	56·1	7·5
13½ – 9	55·8	4·8	57·7	4·3	60·5	5·9	59·3	5·4	57·2	6·5
9 – 4½	57·5	6·1	58·8	6·5	59·2	4·7	60·6	5·2	58·9	5·4
4½ – 0	57·2	5·3	57·9	4·5	58·2	8·1	65·2	2·8	61·2	3·6

With sizes less than 1½-in., the total porosity of the pieces increased as they approached the grate (and the air-supply) and closed pores were opened. Examination of sections of the 1½—1-in. coke taken from near the grate showed that pores near the surface had become enlarged (see Fig. 31), whilst the coke there had lost its original grey colour. Air had evidently penetrated to a depth of about ⅛-in. The increase in porosity of the smaller sizes of coke near the grate, as indicated in Table LVIII, was therefore due to the penetration of air. With the larger sizes, the same degree of penetration would represent a smaller proportion of the bulk, so that the effect on the total porosity would be barely noticeable.

The general conclusion is that for pieces of coke larger than about ¼-in., the size, and therefore the external surface-area, is of greater importance than the total surface-area (*i.e.* including pore-surface) in determining their rates of combustion.

Although ordinary cokes are barely permeable to air under normal conditions of combustion, this does not appear to be so with carbonised fuels which have not passed through a plastic state. For example, retort-carbon, taken from the walls of a coke-oven and broken to 1½–1-in. size, was burnt in a furnace of 1 sq. ft. grate-area, with a fuel-bed 1 ft. deep, at a rate of 39 lb. per hour. The specific gravity and porosity of samples taken from near the grate were determined, and are compared with the values for the original retort-carbon in Table LIX.

TABLE LIX.—INCREASE IN POROSITY ON BURNING RETORT CARBON.

Fuel	Specific Gravity				Porosity, per cent.			
	Apparent		Real		Total Pores		Closed Pores	
	Original	Burnt	Original	Burnt	Original	Burnt	Original	Burnt
Retort-carbon .	1·79	1·21	2·00	1·84	14·7	34·2	2·5	10·1
Coke No. 17Q .	0·88	—	1·77	1·93	54·8	59·7	17·5	7·9
Coke No. 13 P(*b*)	0·88	—	1·85	1·97	55·6	62·4	11·8	9·7

Coke No. 17Q was made from a South Yorkshire coking slack in an 18-in. oven, coked in 36 hours. Coke No. 13P(*b*) was coke from a "box test" made by blending 5 per cent. of coke dust with Parkgate slack.

The porosity of the retort-carbon was doubled and its specific gravity greatly reduced. With the two cokes, on the other hand, the specific gravity was increased. The changes in the retort-carbon can be regarded as indicating that it had a higher specific reactivity after passing through the fuel-bed than when it was charged, whereas the increase in the real specific gravity of the cokes would make them less specifically reactive. The retort-carbon, under the conditions of test, proved to be as reactive a fuel as the cokes; the maximum temperature attained was 1750° C.; oxygen had been removed from the blast within 4 in. of the grate; and the products of combustion contained 20 per cent. of carbon monoxide within 9 in. of the grate. In contra-distinction, in small-scale laboratory tests, retort-carbon has always been found to be "unreactive."

It can be supposed that a fuel might become increasingly permeable to air during its combustion and, in consequence, would become more reactive. The effect on its reactivity of the permeability of a fuel is well illustrated by the performance of wood charcoals, which are considerably more reactive than cokes and have been shown by Bardenheuer and Thanheiser (*Mitt. Kaiser-Wilhelm Inst. für Eisenforschung*, 1924, **5**, 13) to be much more permeable to gases. Meunier (*Chim. et Ind., Numéro Spécial,*

1934, **31**, 602) found, in laboratory tests, that the reactivity of five cokes with carbon dioxide at 950° C. was directly related to their permeability. He also concluded that the reactivity of the cokes was independent of their size. The range of sizes used by Meunier, however, was 10–8, 8–5, 5–3 and 3–1 mm. Had he used larger sizes, it is unlikely that he could have drawn such a conclusion.

So far as metallurgical cokes are concerned, their permeability can rarely affect their performance during normal combustion,

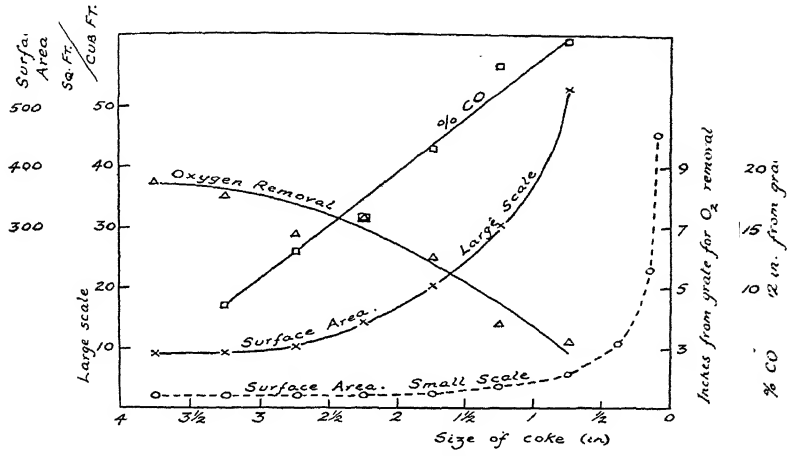


FIG. 32.—The Effect of Size of Coke on the Reactions in a Fuel Bed. The Relationship between the Surface Area of Coke and the Disappearance of Oxygen.

unless pieces less than about ¼-in. in size are used, for it has been shown that their penetration by oxygen during combustion does not exceed about ⅛-in. That, in consequence, the amount of external surface is the factor upon which the reactivity of cokes of the sizes commonly used in practice mainly depends is illustrated by Fig. 32, which records the distance from the grate at which the oxygen of an air-blast was removed when coke of different sizes was burnt under standard conditions in a furnace 2 ft. in diameter with a 2-ft. fuel bed. Distances for the removal of oxygen from the blast are plotted against external surface-areas of the cokes.

Fig. 32 also records the percentages of carbon monoxide found in the gases 12-in. above the grate with the different sizes of coke. The theoretical maximum amount of carbon monoxide that could be present with a dry air-blast is about 34·5 per cent. By extrapolation of the curve in Fig. 32, it appears that this amount would be obtained with coke of ½-in. mean size, whilst it is seen that with coke of 4–3½-in. size the amount was only 3 per cent. The curve relating the production of carbon monoxide at a given zone in the

fuel-bed with external surface-area of the coke is nearly a straight line, there being no point of inflexion as in the curve for the removal of oxygen. The probable reason for this is the endothermicity of the reaction, which lowers the temperature of the fuel-bed. For example, the percentage of carbon monoxide in the gases at zones $7\frac{1}{2}$ in. and 12 in. above the grate with coke No. 13H were :

Size of Coke, in.	$3\frac{1}{2}$–3	3–$2\frac{1}{2}$	2–$1\frac{1}{2}$	$1\frac{1}{2}$–1	1–$\frac{1}{2}$
At $7\frac{1}{2}$-in. CO per cent. . .	1·5	16	15·1	20	27·5
At 12 in. CO per cent. . .	10	22	25·5	30	31

Had the rate of reduction of carbon dioxide not slowed down above the level $7\frac{1}{2}$-in. from the grate, the coke of 1–$\frac{1}{2}$ in. size, for example, should have yielded the maximum possible amount of carbon monoxide (34·5 per cent.) at a distance of 9-in. from the grate. The absorption of heat by the reaction, however, has gradually lowered the temperature of the fuel-bed until, above the zone $7\frac{1}{2}$-in. from the grate, the rate of the reaction is materially reduced.

The reactions $C+O_2=CO_2+174{,}800$ B.Th.U. and $CO_2+C=2CO-70{,}200$ B.Th.U. are not affected to the same degree by the conditions within the fuel-bed. Above the ignition-temperature, say over 700° C., the oxidation of carbon, being an exothermic reaction, is controlled not so much by the temperature as by the rate of supply of oxygen and the area of reacting carbon surface. Kreisinger, Ovitz and Augustine (*U.S. Bureau of Mines Tech. Paper No.* 137, 1916 ; see *Fuel in Science and Practice*, 1935, 271, 296, 331, 364 ; 1936, 16, 59) found that, for a rate of combustion of 120 lb. per sq. ft. of grate-area per hour (when the maximum temperature was 1600° C.), the oxygen of the air-blast was completely removed by a 6-in. bed of coke of $1\frac{1}{2}$-1 in. size. Our own tests, which are described later, show that with a rate of combustion as low as $1\frac{1}{2}$ lb. per sq. ft. of grate-area per hour (the maximum temperature being 800-900° C.), all the oxygen of the air-blast had disappeared within a few inches of the grate. With a given rate of supply of air, the distance from the grate at which the oxygen is used up depends mainly on the amount of carbon surface exposed to the blast. With most cokes, as has been shown, the amount of reactive surface is proportional to the size of the pieces.*

The reaction between carbon dioxide and carbon, on the other hand, is mainly dependent on the temperature. In the main stream of gases within a fuel-bed, carbon monoxide cannot remain

* When the pieces are less than about $\frac{1}{4}$-in. in size, and in certain circumstances when larger pieces are used, the porous structure of the coke may be more effective then usual in providing reactive surface, and the oxygen in the air-blast may be used up earlier than would be expected. It is possible that the small differences recorded in our first report for the reactivity with oxygen of a number of cokes of $1\frac{1}{2}$–1-in. size, burnt at the rate of 39 lb. per sq. ft. of grate-area per hour, can be ascribed to differences in the effective surface that they presented.

so long as oxygen also is present, and the distance above the grate at which it can be collected in the products of combustion depends upon the distance needed for the complete removal of oxygen from the air-blast. As soon as carbon monoxide begins to accumulate in the products of combustion, the temperature of the fuel-bed begins to fall and the rate of reduction of carbon dioxide also begins to fall. Since with the smallest sizes of coke the oxygen in the air-blast is used up soonest, carbon monoxide appears earlier in the products of combustion and the temperature of the fuel-bed above the oxidation zone falls more quickly than when larger pieces of coke are used. The maximum temperature of combustion for a given rate of supply of air does not vary much with different sizes of a coke, but the temperature rises rapidly as the rate of supply of air is increased. The higher the maximum temperature of the fuel-bed obtained by combustion of the coke, the longer can the endothermic reduction of carbon dioxide be maintained.

We must, therefore, recognise a marked contrast between the two principal reactions concerned in the combustion of a bed of coke by an air-blast. The rate of reaction of oxygen with the coke is determined mainly by the amount of reacting surface available; the rate of reduction of carbon dioxide, although influenced by the amount of reacting surface, is determined mainly by the temperature.

Experimental Work

The experimental work that we have carried out on the combustion of coke has been with furnaces 2-ft., 13½-in. and 8-in. in diameter, burning coke from ½-in. upwards in size.

The 2-ft. diameter furnace was used mainly to determine the effect of the size of coke on its combustion and was similar in design to the 13½-in. diameter furnace described in our first report. It is illustrated in Fig. 33. The wind-box and the casing of the furnace are of ⅛-in. welded steel plate. An ash-pit door and a charging-door are fitted in such a manner as to be gas-tight. The casing is lined with shaped silica brick, which had been assembled when "green" and hard-burned *in situ*. Sampling nipples are welded to the casing to provide sampling-points at 1½-in. intervals above the grate up to a 24-in. level, and there are sight-holes, in addition, at 1-in. and 2-in. levels. The volume of air supplied to the furnace is measured by a Venturi gauge.

When carrying out a test, a coke was broken or screened to yield sizes 4–3½, 3½–3, 3–2½, 2½–2, 2–1½, 1½–1 and 1–½-in. square mesh by hand-screening. The larger sizes, over 3-in., were obtained without breakage. The bulk-density of the coke was measured in a box of 2 cu. ft. capacity and the quantity required to provide a bed 24 in. deep in the furnace was determined. The coke was ignited under natural draught by a gas-burner and the air supply was then adjusted to the amount necessary for the

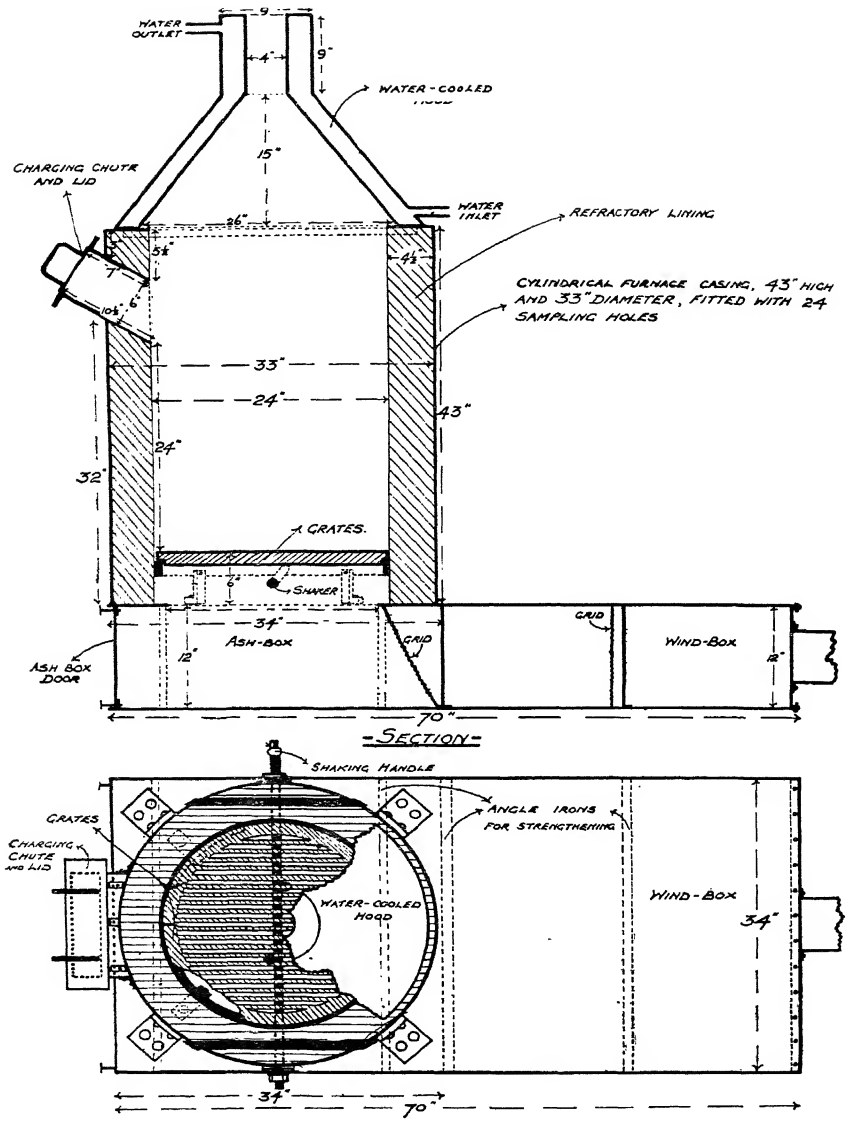


FIG. 33.—" Combustibility " Furnace, 24-in. Diameter.

rate of combustion desired. When the fuel-bed glowed red throughout and gas was burning at the cowl of the furnace, the test was begun.

A test was continued during 3 hours to ensure that, during the last hour, the lower 12 in. of the original coke-bed had been completely burnt away, and that the portion of the bed then examined had undergone reaction within the upper 12 in. of the furnace. Gas-samples were taken from 8 or 10 of the sampling-points through water-cooled pipes of copper at 1½ and 2½ hours from the beginning of the test, and temperatures were read, by means of a disappearing-filament pyrometer, at 1, 2 and 3 hours. The furnace was fed with coke at convenient intervals so as to maintain a depth of fuel-bed of 2 ft. At the end of the test, the air-blast was cut off and the furnace allowed to cool; the charge was then emptied out and the ash and clinker examined and weighed.

Three Yorkshire cokes were tested in this manner, namely:

No. 79. From Haigh Moor and Barnsley coals coked in 20-in. ovens in 24-34 hours.

No. 13*H*. From Parkgate coal coked in 20¾-in. ovens in 28 hours.

No. 31*C*. From Halifax Soft coal coked in beehive ovens.

Some of the properties of these cokes are recorded in Table LX.

TABLE LX.—PROPERTIES OF COKES USED FOR COMBUSTION TESTS.

Coke No.	Shatter Indices		Specific Gravity		Total Porosity %	Analysis of dry Coke. Per cent.			Melting Point of Ash, °C.*
	1½-in.	½-in.	Apparent	Real		Ash	Vol. Matter	Sulphur	
79 .	83	97·4	0·92	1·84	50·0	8·6	1·9	1·04	1290
13 H .	84	98·0	0·88	1·79	52·7	9·6	1·2	1·40	—
31 C .	93	98·4	0·88	1·83	51·8	5·6	0·2	0·71	1325

* In an oxidising atmosphere.

When testing coke No. 79, the air-supply used was the maximum available, namely, 104 cu. ft. per minute measured at 60° F. and 30 in. mercury. As any test proceeded, and clinker accumulated in the furnace, the resistance of the fuel-bed to the passage of air increased and the rate of supply of air tended to fall, as is shown by the records in Table LXI.

TABLE LXI.—EFFECT OF SIZE OF COKE ON RATE OF SUPPLY OF AIR.

Size of Coke, in. . . .	4–3½	3½–3	3–2½	2½–2	2–1½	1½–1	1–½
Bulk density, lb. per cu. ft. .	24	25	26	27½	27½	29	31
Initial air-blast, cu. ft. per min.	104	102	104	104	102	100	98
Final air-blast, cu. ft. per min. .	104	100	102	102	100	93	91

TABLE LXII.—COMBUSTION OF COKES OF DIFFERENT SIZES. TWO-FT. DIAMETER FURNACE.

Coke No.	Size of Coke, in., sq. mesh								
	4–2	3–2	4–3½	3½–3	3–2½	2½–2	2–1½	1½–1	1–½
	Bulk-density of coke in furnace, lb. per cu. ft.								
79 .	—	—	24	25	26	27½	27½	29	31
13 H .	—	—	—	26	26	27½	29	31	32
31 C .	27½	27½	26	26	26	24	29	29	31
Mean :			25	25·7	26	26·3	28·5	29·7	31
	Average rate of combustion, lb. per sq. ft. of grate-area per hr.								
79 .	—	—	15	16½	19	21½	22	25	22
13 H .	—	—	—	13½	17	14	19½	18	20
31 C .	18	17	—	15	15½	17	18	24½	23
Mean :			15	15	17·2	17·5	19·8	22·5	22
	Average distance from grate at which oxygen disappeared, in.								
79 .	—	—	9	7½	7½	8	6	3	3
13 H .	—	—	—	7½	7	6	6	4	4
31 C .	6	9	8*	9	6	8	6	4½	3
Mean :			8·5	8	6·8	7·3	6	3·8	3·3
	Average carbon monoxide in gases 12 in. above grate, per cent.								
79 .	—	—	2·8	8·8	13·0	13·1	21·7	29·2	31·4
13 H .	—	—	—	10·5	12·5	21·0	22·0	29·5	31·5
31 C .	13·1	15·1	7·6*	6·5	13·7	13·0	21·3	26·8	29·3
Mean :			—	8·6	13·1	15·7	21·7	28·5	30·7
	Maximum temp., 3 hr. after beginning of test, ° C.								
79 .	—	—	1550	1590	1650	1560	1630	1630	1570
13 H .	—	—	—	1580	1620	1520	1590	1610	1520†
31 C .	1580	1640	1570	1560	1610	1580	1620	1640	1600
Mean :			1560	1577	1623	1533	1613	1627	1563
	Distance from grate at which max. temp. was attained, in.								
79 .	—	—	6–7½	4½	3–4½	6	1½–3	3	1½
13 H .	—	—	—	3–4½	4½	6	3	3	1½
31 C .	4½–9	1½–3	7½	7½	4½–6	3	3	3	1½
Mean :			7½	5	4½	5	3	3	1½
	Average temp. in lower 15-in. of bed 3 hr. after beginning of test, ° C								
79 .	—	—	1488	1507	1567	1472	1496	1468	1347
13 H .	—	—	—	1492	1517	1411	1485	1413	1365
31 C .	1509	1532	1519	1520	1533	1468	1496	1471	1405
Mean :			1503	1506	1539	1450	1492	1451	1372

* A bed of small coke was necessary for ignition.
† At No. 1 sampler. The temperature may have been higher below this point.

With the $1\frac{1}{2}$–1-in. and 1–$\frac{1}{2}$-in. sizes, of high bulk-density, the initial rate of supply of air was less than with the larger sizes and, since the clinker produced by them was resistant to the passage of air, the rate fell off rapidly.

In later tests, the initial rate of supply of air was reduced to 88 cu. ft. per minute, and the air-valve was opened as the test proceeded to overcome increasing resistance of the fuel-bed. The static pressures needed to force the required quantities of air through the furnace when different sizes of coke No. 31 C were used were :

Size of Coke, in.	$3\frac{1}{2}$–3	3–$2\frac{1}{2}$		1–$\frac{1}{2}$
Pressure, in. W.G.—				
at beginning .	0·2	0·2	0·2	0·5
after 1 hr. .	0·2	0·25	0·2	0·5
after 2 hr. .	0·25	0·35	0·45	0·65
after 3 hr. .	0·35	0·35	0·5	0·75

A summary of the principle data obtained during tests with the three cokes in the 2-ft. diameter furnace is given in Table LXII.

It will be seen that, despite the precaution taken to maintain a constant rate of supply of air, the rate of combustion of the smallest size of coke showed a tendency to decline. This may have been due to leakage of air at the ash-pit door under the higher pressure required to force the blast through the bed of small coke.

The rapid diminution of the distance from the grate at which the oxygen of the air-blast is used up, so soon as the size of the coke falls below 2-in., has already been discussed (see Fig. 32). At the zone in the bed where oxygen disappears, the amount of carbon dioxide in the gases is at a maximum or has just begun to fall ; whilst the zone of maximum temperature is just below that for removal of oxygen. Fig. 34 shows the percentages of carbon dioxide found in the gases at different levels of the bed for coke No. 79 of different sizes. For the sizes over 3-in., there is but little reduction of carbon dioxide in 12-in. of the bed, but, with sizes under $1\frac{1}{2}$-in., reduction is nearly complete within the same distance.

By reason of the marked reduction of carbon dioxide when the smaller sizes of coke are used, the temperature of the upper part of the fuel-bed falls rapidly, whereas with the largest size the fall in temperature is slight (see Fig. 35). Although with the larger sizes of coke the maximum temperatures attained within the fuel-bed are not as high as with the smaller sizes, the average temperature remains higher because there is less reduction of carbon dioxide.

A notable feature of the tests was the variation in the formation of clinker with the size of coke used. The melting-points of the ashes of cokes Nos. 79 and 31C were fairly high, namely 1290° C. and 1325° C. With the larger sizes of these cokes the clinker produced was granular and fragile, and most of it dropped between

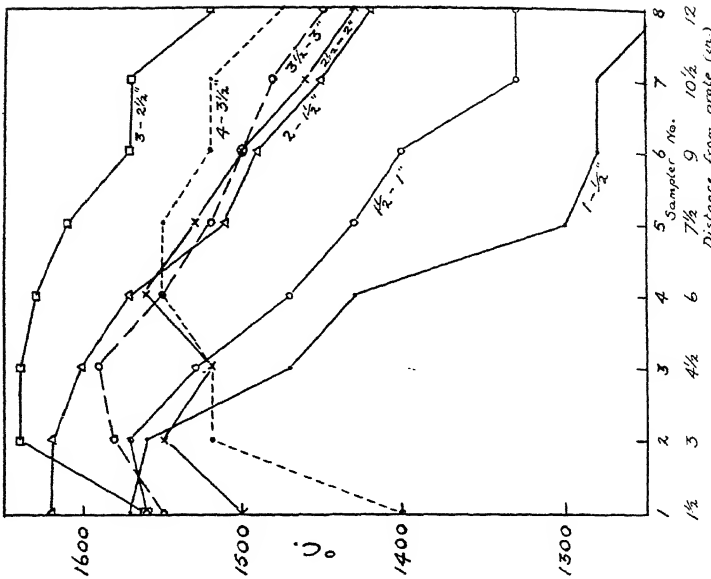
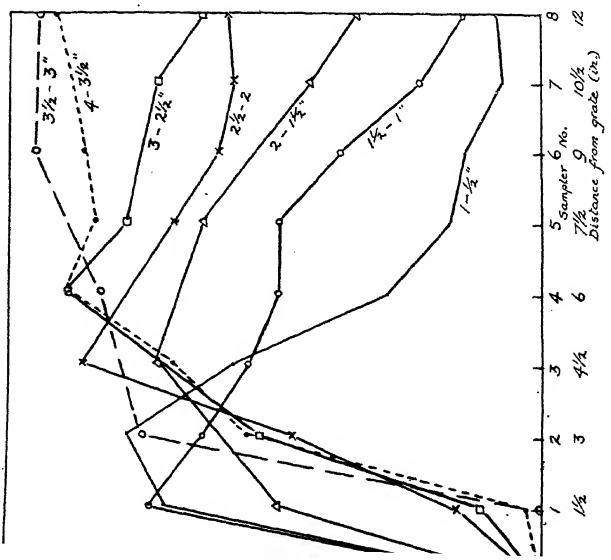


FIG. 35. The Temperatures at Different Levels of a Fuel Bed with Different Sizes of Coke (No. 79).



—Carbon Dioxide at Different Levels of a Fuel Bed with Different Sizes of Coke (No. 79. Average for two analyses).

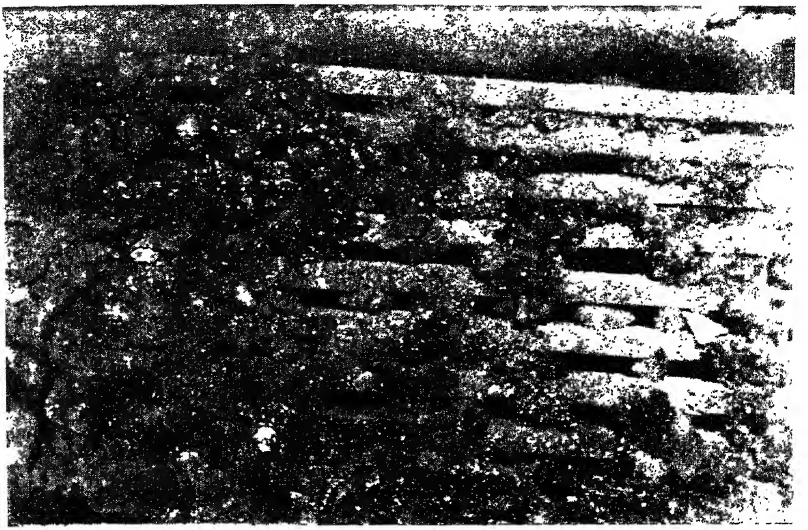


FIG. 36.—Large Coke.

FIG. 37.—Small Coke.

The Difference in Character of Clinker on Grate Bars when Burning Large and Small Coke under the same Conditions.

the grate-bars, but with the smallest size a massive sheet of clinker formed over the bars and offered high resistance to the blast. On cooling, the layer of clinker was found to have flowed between the grate-bars from which a layer of scale was detached on its removal. The proportions of the ash that remained as clinker on the bars (being over $\frac{1}{2}$-in. in size) with coke No. 31C of different sizes, were:

Size of Coke, in.	$3\frac{1}{2}$–3	3–$2\frac{1}{2}$		2–$1\frac{1}{2}$		
Clinker over $\frac{1}{2}$-in. in size (per cent. of total ash)	27	49	59	60	59	59

The difference in the character of the clinker obtained with different sizes of the same coke is shown by Figs. 36 and 37, which are photographs of the clinker resting on the grate. The clinker from the larger coke was in small aggregates, which did not cover the bars, whilst that from the smaller coke covered the bars completely. With intermediate sizes of coke the clinker was intermediate in character. The character of the clinker obtained with the small coke was no doubt due in part to the high temperature near the grate-bars, and in part to the extensive zone of the coke-bed in which reducing conditions prevailed. The reduction of iron in the ash from the ferric to the ferrous condition would lower its fusion temperature. An additional cause of the troublesome clinker with small coke is the inability of ash to fall between the small voids in the bed without coming into contact with coke.

Reactions within a Burning Bed of Coke

From the results of the tests with different sizes of coke and a standard rate of supply of air, an analysis can be made of the reactions within a burning bed of coke.

The amount of coke burnt within the oxidation zone is almost constant, irrespective of the size of the pieces. It is improbable that, with rapid rates of combustion, the reaction $C + O_2 = CO_2$ is completed, because dissociation of carbon dioxide can occur at the high temperatures attained. Out of 300 analyses of the gases drawn from the combustion zone when the rate of combustion of the coke exceeded 13 lb. per sq. ft. of grate-area per hour (the usual rate being 39 lb.), only 11 contained more than 18 per cent. of carbon dioxide and the maximum quantity was 18·3 per cent. Theoretically, for an average coke, about 20·5 per cent. of carbon dioxide would be expected in the products of combustion. The dissociation of carbon dioxide involves a loss of heat.

Heat is lost also by radiation from the furnace. The amount so lost is proportional to the area of the radiating surface, and is greater with the larger sizes of coke because of the greater extent of the higher-temperature (oxidation) zone. An approximate measure of the variation in the amount of heat lost by radiation

with different sizes of coke can be obtained on the assumption that the loss is proportional to the weight of coke within the oxidation zone. In Fig. 38 this assumption is made, and values are also given for the external surface-areas of the different sizes of coke required for the burning of 1 lb. per sq. ft. of grate-area per hour. For the

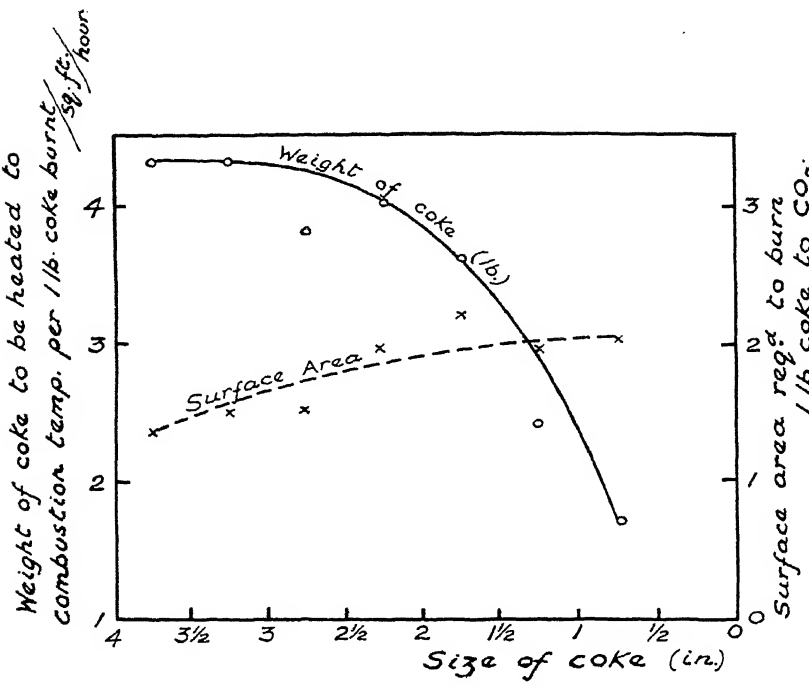


FIG. 38.—Comparison of the Weight and Surface Area of Different Sizes of Coke for the Combustion of 1 lb. per sq. ft. per hr.

last-named values the surface areas of the pieces of coke within the oxidation zone have been calculated, a layer of 1 in. over the grate being excluded from the oxidation zone as being inactive, either through accumulation of ash or because it serves to preheat the air. It will be seen that the area is almost constant for the sizes between $2\frac{1}{2}$–2 in. and 1–$\frac{1}{2}$ in., but is less for the larger sizes. The inference is that for a given rate of consumption of coke, combustion on the surface of the larger sizes is more intense than with the smaller sizes, and that, if the external heat-loss were constant,

they would provide the higher combustion temperatures. It is probable, therefore, that in a blast-furnace or cupola, where the external heat-loss is considerably less than in an experimental furnace, large coke produces as high combustion-temperatures as small.

The temperature of combustion of coke can be calculated from the formula $T = \frac{H - h_d - h_e}{S}$, where T is the temperature (in ° F.), H is the total heat of combustion, h_d is the heat lost by dissociation of carbon dioxide, h_e is the external heat-loss in B.Th.U. per lb. of coke burnt per hour, and S the specific heat of the products of combustion of 1 lb. of coke. When 1 lb. of coke of 90 per cent. carbon content is burnt with air completely to carbon dioxide, the products of combustion have a specific heat of 2·75 B.Th.U. per ° F., and the theoretical temperature of combustion (there being no loss of heat) is 4,700° F. (2,570° C.).

Reactions in the Hearth of a Blast-Furnace.—In a blast-furnace, the principal product of combustion over most of the width of the hearth is carbon monoxide, because the oxidation zone occupies but a small proportion of the area of the hearth, and the heat of reaction is 3,920 B.Th.U. per lb. of coke of 90 per cent. carbon-content. The theoretical temperature of combustion of coke to carbon monoxide is 1425° C. or, if the air is preheated to 645° C. (1200° F.), 1975° C. Since a temperature of over 1600° C. is required to complete the hearth reactions and to ensure free running of the slag, it is easy to see why the use of a large hearth was impracticable with a cold blast, and why it was usual to narrow the hearth to as little as 2 ft. With a narrow hearth, there would be a concentration of the oxidation zone (of theoretical temperature 2570° C.), in which the final fusion of the iron and the slag would be completed. In a modern blast-furnace using pre-heated air, the theoretical temperature when carbon burns to carbon monoxide may be as high as 1980° C., and concentration of the oxidation zone is unnecessary.

Reactions in a Cupola.—Whereas in a modern blast-furnace with a wide hearth the chief product of reaction of oxygen with carbon is carbon monoxide, in a cupola the high-temperature oxidation zone is larger in proportion and it is important that it should be as large as possible because a cold air-blast is used. For this reason, an unreactive coke in large pieces is preferred for cupola work. With a highly-reactive coke its oxidation to carbon dioxide and the reduction of carbon dioxide to carbon monoxide may occur almost simultaneously, thus decreasing the volume of the high-temperature zone.

The extent to which the maximum combustion-temperature

is affected by the rate of combustion of the coke (or the rate of supply of air) is illustrated by the results recorded in Fig. 39.

Some of these results were obtained in the 13½-in. and 24-in. diameter furnaces previously described, and others in a furnace of 8-in. diameter. In the construction of this 8-in. furnace (Fig. 40), an ⅛-in. casing of mild steel is lined with rammed sillimanite to a thickness of 2 in. A grate is supported 7 in. above the base, the grate bars being castings designed to provide point contact for the

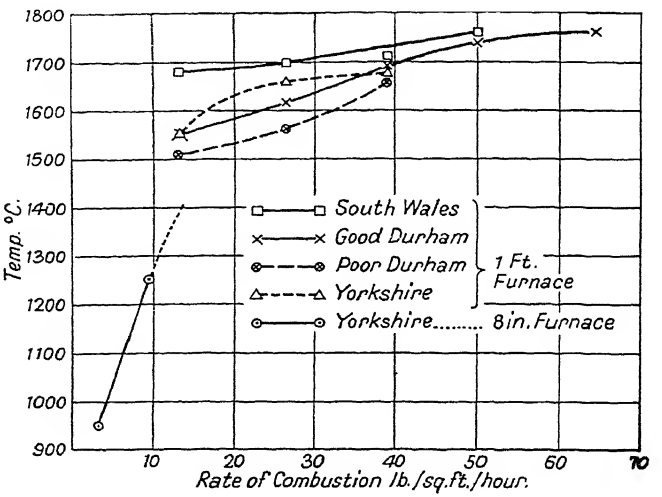


FIG. 39.—The Effect of Rate of Combustion on the Maximum Combustion Temperature in a Fuel Bed of Coke of Standard Size.

fuel-bed, so that the air-blast shall keep them cool. The space between the bars can be either ½-in. or ¾-in. A 12-in. bed of coke can be used with a 6-in. air-space above it, the top of the furnace, like the base, being a standard fitting for a slow-combustion stove. Air is supplied through a 1-in. main, 10 ft. long, fitted with a Pitot tube for the measurement of quantity. Bosses are welded to the casing to provide for sampling-points 1, 2, 3, 5, 6, 7½, 9 and 12 in. above the level of the grate. Sampling-tubes of fused silica are used, of ⅛-in. internal diameter with thick walls. These are air-cooled. The gases from the fuel-bed can be drawn by an aspirator through a series of connecting-tubes to a central collecting-point, where a portion of the gas-stream is by-passed and collected over mercury.

The results of three series of tests are illustrated in Fig. 41, namely, for (1) 1½–1-in. coke burnt in a 13½-in. furnace with a 12-in. fuel-bed at rates of 65, 39 and 12 lb. per sq. ft. of grate area

per hour; (2) $1\frac{1}{2}$-1-in.* coke burnt in furnaces of 24, $13\frac{1}{2}$ and 8 in. diameter with a 24-lb. rate of combustion, using a 24-in. bed with the 24-in. furnace and 12-in. beds with the others; and (3) coke burnt at rates of 10, $3\frac{1}{2}$ and $1\frac{1}{2}$ lb. in the 8-in. furnace with a 12-in. fuel bed.

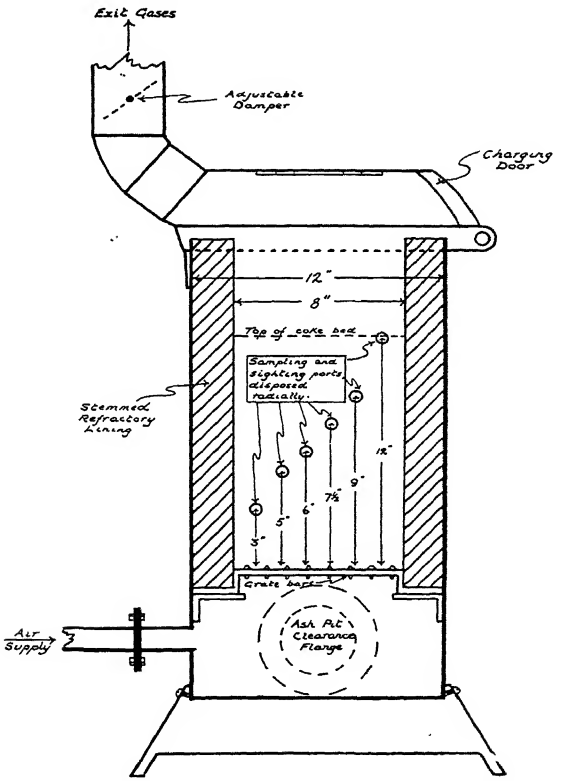


FIG. 40.—"Combustibility" Furnace, 8-in. Diameter.

The first series of tests shows that the maximum temperature of combustion falls with decreasing rate of combustion. Comparison of Series 1 and 2 shows that the maximum temperature was influenced to a greater degree by the diameter of the furnace, *i.e.* by the external heat-loss, than by the rate of combustion. In Series 3, the temperature fell rapidly with the rate of combustion because of the high external heat-loss. The effect of a fall in the temperature of combustion can be seen by comparing Tests 1

* $1\frac{1}{4}$ to $\frac{3}{4}$-in. size in the furnace of 8-in. diameter.

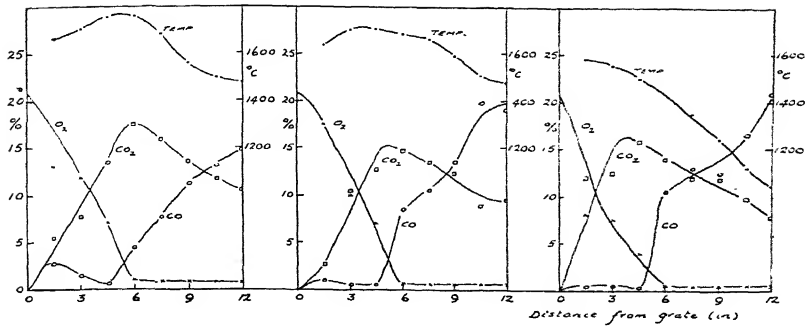
and 2 in Series 3. In Test 1 of this series, with a rate of combustion of 10 lb. and a maximum temperature of 1200° C., the concentration of carbon monoxide reached a value of 20 per cent., a result comparable with those of Tests 2 and 3 of Series 1 save that, because of the longer time of contact, 20 per cent. of carbon monoxide was found at a lower level in the fuel-bed in Series 3. When the rate of combustion was reduced to 3½ lb., the maximum temperature was only about 1000° C., and the amount of carbon monoxide formed decreased in consequence.

Another effect of temperature is illustrated by Tests 1 and 2 of Series 2. In Test 1, a 24-in. bed was used and the temperature at a level 12 in. above the grate was as high as 1400° C.; but when, in Test 2, a 12-in. fuel-bed was used, the temperature at the 12-in. level dropped to 1000° C. owing to radiation from the upper surface of the bed. The maintenance of a high temperature 9–12 in. above the grate in the 24-in. bed enabled the carbon monoxide content of the furnace-gases to continue to increase, but in Test 2 no such increase occurred.

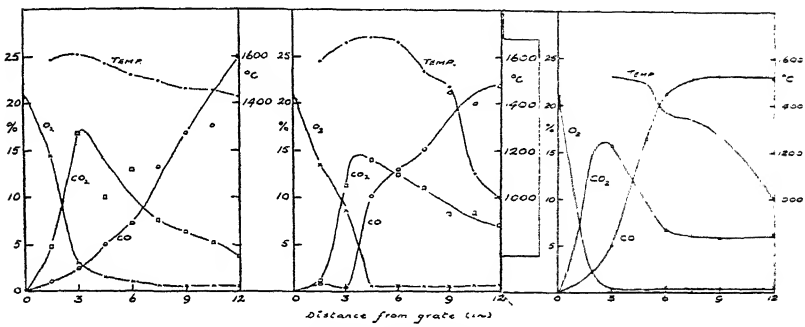
The maximum temperature in the fuel-bed was reached in these tests when, or slightly before, the furnace-gases contained the maximum amount of carbon dioxide, the oxygen in the blast having all disappeared. Although a little carbon monoxide was frequently found within 1½-in. of the grate, the amount invariably decreased as the fuel-bed was ascended to the point where oxygen had disappeared, a stage signalised by the reappearance of carbon monoxide. The extent to which the carbon dioxide formed in the early stages of combustion was reduced higher up in the fuel-bed depended upon the temperature at which the gases entered the reduction zone. With slow rates of combustion and high external heat-losses but little reduction occurred.

Perhaps the most significant observation from all these tests is the removal of oxygen from the blast within 6 in. of the grate with coke up to 1½-in. in size, even though the rate of combustion was varied from 1½ to 65 lb. per sq. ft. of grate-area per hour. Kreisinger has extended this observation to rates of combustion of 120 lb., and it can be deduced that the rate of combustion is mainly determined by the rate of supply of air equally in the domestic boiler (1 to 6 lb. per sq. ft. of grate-area per hour) and in the blast-furnace (75 to 120 lb.).

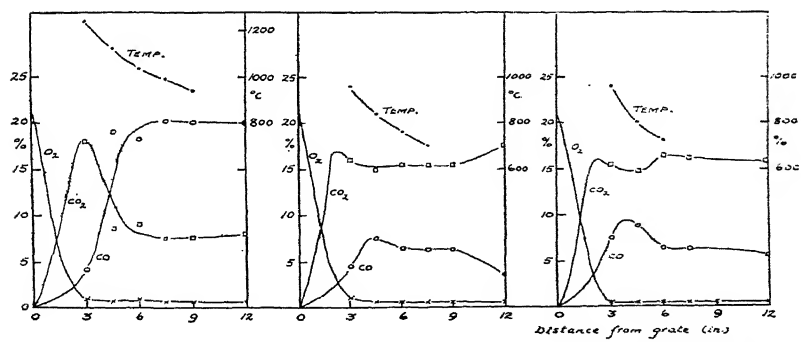
It would seem that, in a blast-furnace, high hearth-temperatures should be promoted (1) by reducing the external heat-loss per lb. of coke burnt (by increasing the diameter of the hearth), and (2) by enlarging the oxidation zone (by ensuring a low density of packing of coke near the tuyeres and excluding, as far as possible, coke less than 1½-in. in size). In illustration of the effect of (1), Marshall's determinations of the external heat-loss of blast-furnaces (*Journ. Iron and Steel Inst.*, 1935, **I**, 59) can be expressed in the following form :



Series 1.—Effect of Rate of Combustion in 13½-in. Furnace: 65, 30 and 13 lb./sq. ft./hr.



Series 2.—Effect of Size of Furnace Burning 24 lb./sq. ft./hr.; 24, 13½ and 8-in. Diameter Furnaces.



Series 3.—Effect of Low Rates of Combustion in 8-in. Furnace; 10, 3½ and 1½ lb./sq. ft./hr.

FIG. 41.—The Effect of Rate of Combustion and Size of Furnace on the Reactions in a Fuel Bed.

Furnace	Production of Iron. Tons per week	External Heat Loss		Theoretical Combustion Temp. ° C.	
		B.Th.U. per lb. of coke burnt	Per cent. of total heat of combustion of 1 lb. of coke *	Allowing for external heat loss	Assuming no external heat loss
C	2,800	170	3	1810	1860
D	1,600	315	7	1690	1785
B	990	495	10	1640	1810
A	750	638	12	1735	1950
B	550	1,040	19	1640	2010

* Coke of 90 per cent. carbon burning to carbon monoxide, with sensible heat added.

At the furnaces producing less than 1,000 tons of iron per week, the external heat-loss in the hearth zone represents a high proportion of the heat released in the hearth and has a considerable influence on the theoretical temperatures.

The Combustion of Coke in Steam Boilers.—In areas remote from the coalfields, gas-works coke may be cheaper than coal, and considerable use has been made of it as a steam-boiler fuel. The results of our tests with the 24-in. furnace show that the formation of clinker might be troublesome if coke of less size than 1½-in. were used for this purpose, and that 3–2 in. size would be more suitable, for with the larger coke the zone of maximum temperature during combustion is removed further from the grate-bars.

Trouble has sometimes been experienced, when using coke for steam-raising, through the burning of the grate-bars. This has usually been because the bars used have been designed for burning coal. When using coke, the bars should preferably be thin and tapered in such a way as to provide ample air-cooling. The London and Counties Coke Association recommend bars with 50 per cent. air-spacing, the spaces to be at least ¾-in. wide. Cooling of the bars can also be effected by admitting steam beneath them.

The effective combustion of coke on a boiler grate is simpler than that of coal, because of the almost complete absence of volatile matter, and primary and secondary air-supplies can readily be adjusted. The transmission of heat by radiation from a bed of coke is high, so that rapid rates of evaporation of water and high thermal efficiencies can be obtained.

The Combustion of Coke in Central-Heating and Domestic Boilers.—Manufacturers of central-heating boilers for coke design them for combustion at slow rates, so as to avoid high temperatures in the fuel-bed and thus to minimize the formation of clinker. Such boilers can be left unattended over long periods. Data with respect to central-heating boilers of different sizes are given in Table LXIII.

TABLE LXIII.—CENTRAL-HEATING BOILERS. DIMENSIONS AND RATING.

Designation	Dimensions (in.)	Grate Area (sq. ft.)	Depth of Fuel Bed (in.)	Fuel Capacity (lb.)	Rate of Combustion (lb. per sq. ft. of grate per hr.)	Heating Surface (sq. ft.)	Rating, B.Th.U. per hr. (× 1000)
Domestic .	9 × 9	0·6	10	12	6·2	2	—
No. 3 .	12 × 11	0·9	16	28	4·0	10½	46
No. 6 .	16 × 15	1·7	22	78	6·5	26	114
18K .	44 × 12	3·9	19	155	5·6	43	159
29K .	50 × 22	6·6	19½	370	5·7	83	364
310K	66 × 32	11·2	22	324	6·5	145½	643
410K .	66 × 42	14·2	22	658	6·4	204	904
413K .	87 × 42	19·0	22	880	6·5	267	1,183

The fuel-capacities in Table LXIII are based on the use of coke of bulk-density 25 lb. per cu. ft. The "ratings" represent the quantities of heat, in B.Th.U. per hour, that can be dissipated by hot-water radiators connected to the boilers, and are calculated on the basis of heat-transmission from the coke-fire to the water in the boiler at a rate of 4,400 B.Th.U. per sq. ft. of heating surface per hour. Assuming a thermal efficiency of about 75 per cent. (at the stated "rating"), the fuel-capacity of the boilers is such that refuelling is needed every six hours, one-sixth of the charge remaining at the end of that time to provide ignition of a fresh charge.

It is noteworthy that the same rate of combustion, about 6 lb. per sq. ft. of grate area per hour, is specified irrespective of the size of the boiler. Since a central heating system is usually designed to maintain room-temperatures at 60° F. when the outdoor temperature is 32° F., and the mean outdoor temperature in Great Britain during winter is about 43° F., the rate of combustion can usually be less than that specified. For example, with an outdoor temperature of 55° F. the rate of combustion would need to be only from ½ to 1 lb. per sq. ft. of grate-area per hour to maintain a room temperature of 60° F.

Central-heating boilers (and domestic hot-water boilers) should operate continuously, day and night, and should therefore be capable of burning without attention (at less than the specified rate of combustion) during at least 8 hours. Most difficulty arises with the smallest boilers used for domestic hot water, some of which hold only about 10 lb. of coke. If, with such a boiler, a period of 8-hours burning unattended during the night is demanded, the rate of combustion must fall as low as 1 lb. per sq. ft. of grate-area per hour, for with such a small boiler it is desirable that at least half the fuel-bed should remain unburnt in order that the fire shall recover on re-fuelling.

The Size of Coke Required.—The primary requirement of a coke for central-heating and domestic boilers is that it should continue to burn with low rates of combustion. At the minimum rate of combustion (say 1 lb. per sq. ft. of grate-area per hour), the settlement of the charge in the fire-pot may be slow, and it is essential that the fuel-bed should consolidate as the lower layers burn away. The smaller the size of the coke the more readily does the charge settle, but, since a fuel-bed of the smallest sizes of coke offers considerable resistance to the passage of air, it may give trouble by clinkering. The size of the coke should therefore not be less than is requisite to provide for steady settlement of the charge. As a rough guide to the appropriate size of coke, the larger screen size should be not more than one-sixth of the smaller dimension of the fire-pot, *e.g.* 2–1½-in. coke for a 12 by 12-in. fire-pot. If the minimum rate of combustion for a central heating boiler is 2 lb. per sq. ft. of grate-area per hour, as it might well be during the colder winter months, a larger coke can be used, say of maximum screen size not more than one-quarter the smaller dimension of the fire-pot, and the use of the larger coke may have advantages. For the larger boilers, however, the coke should not exceed 3 in., for, as shown by the tests previously recorded (Table LVII), coke any larger in size exposes insufficient surface. The best size of coke for a large central-heating boiler is 3–2-in. though the fire may be rather slow in "picking up" should it have been damped down overnight.

In America, where central-heating is more common than in this country, considerable importance is attached, in relation to the formation of clinker, to the fusion-temperature of the ash of the coke. Our tests have shown, however, that clinker-formation depends largely on the size of the coke and on the maximum combustion-temperature, and thus on the rate of combustion (see Figs. 36 and 37).

A low rate of combustion in a central-heating boiler can be ensured by choosing a boiler of ample grate-area for the duty required, and a desire to "force" such a boiler (or a domestic hot-water boiler) need not arise if choice is made of a coke that will stay alight overnight with the minimum rate of combustion. The ability of a coke to remain alight depends not only on its size but on its specific reactivity. On assessing the specific reactivity of a coke from its "critical air blast" (see p. 167), it has been found that cokes for which that determination exceeded a value 0·063 were difficult to burn in small domestic boilers at a rate of 1 lb. per sq. ft. of grate-area per hour during 8 hours, when using coke of 1½–⅞-in. size (round holes). Cokes of higher specific reactivity (lower C.A.B.) than is indicated by that value should therefore be chosen.*

The volume of the ash remaining in a coke boiler influences the rate of combustion of the coke, particularly during burning overnight. The bulk-density of ash from coal shales is about 40 lb.

* Alternatively, a smaller size of coke, say 1⅛–½ in should be used.

per cu. ft., but fine ash of less than 60 mesh may have a bulk-density as low as 10 lb. per cu. ft. The ash obtained on burning coke may thus vary in bulk-density between 10 and about 40 lb. per cu. ft., dependent upon how much of it is derived from coarse shale particles and how much from the ash of finely-ground coal. In a central-heating boiler of 1 sq. ft. grate-area burning 20 lb. of coke during the night, 10 per cent. of ash of the lowest bulk-density would yield a layer about 2½ in. thick. Such a layer would offer considerable resistance to the passage of air and would check the rate of combustion.

The Combustion of Coke in Open Grates.—The bulk-density of sized metallurgical coke is usually about 24 lb. per cu. ft., but it may rise to 28 lb. or more for coke made from compressed charges. To provide a fire which will last a reasonable time in an open grate without need for recharging, about 12 lb. should be used. In a grate 16 in. wide and 8 in. from back to front, this quantity of a coke of average bulk-density would form a fuel-bed about 6½ in. thick. The resistance of the fuel-bed to the passage of air, upon which the maintenance of the fire depends, is affected by the size of the pieces of coke. On the other hand, because metallurgical coke is of low "combustibility" (as compared with coal, for example), it cannot be used effectively in an open grate in large pieces, the upper limit of useful size being about 2-in. If an attempt be made to increase the combustibility of the coke by providing greater surface-area, that is to say, by decreasing the size of the pieces, the fuel-bed may become too resistant to the passage of air. Thus, whilst a 12-in. bed of 1¼–¾-in. size can be used satisfactorily in a closed stove, the draught being about 0·16 in. W.G., coke of that size does not continue to burn satisfactorily in an open grate, because the resistance of the bed is about equal to the chimney-draught.

The effect of the resistance of beds of coke to the passage of air is illustrated by the records reproduced in Fig. 42, which are of the radiation delivered to an area of 1 sq. ft. at a distance of 3 ft. from the front of a grate in which a reactive (low-temperature) coke was burnt under several different conditions. In the first series of tests the coke was burnt on a horizontal grate to which the access of air from beneath the bars was prevented. In the second series, air was allowed to enter beneath the bars. In each series, the radiation records (in Fig. 42) were taken after one charge of coke had burnt down until radiation fell to 200 B.Th.U. per sq. ft. per hour and a second charge had been added.

In both series of tests it was found that the second charge of coke had to be added sooner the larger the size of the pieces, but radiation from the fire increased most rapidly and attained the highest values with the largest pieces. The denser packing of the smaller sizes of the coke apparently checked the passage of air

through the fuel-bed and thus diminished the rate of combustion, despite the greater surface of coke available for reaction. When air was admitted beneath the grate, the radiation reached a high value more rapidly. In this series of tests, the combustion was most intense just above the grate-bars, *i.e.* in the heart of the fire, and there was little, if any, cooling by air passing through the front of the fire. The average surface-temperatures of the fires at maximum

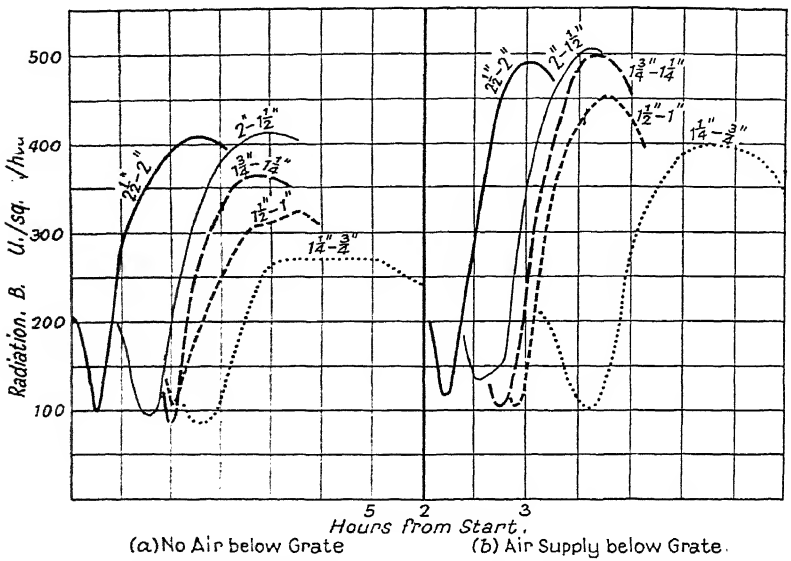


FIG. 42.—The Effect of Method of Air Supply on the Radiation from Different Sizes of Low Temperature Coke burnt in an Open Grate.

radiation were: First Series, 970, 940, 940, 940, 900° C.; and, Second Series, 980, 1000, 1000, 950, 950° C., for coke of $2\frac{1}{2}$–2, 2–$1\frac{1}{2}$, $2\frac{3}{4}$–$1\frac{1}{4}$, $1\frac{1}{2}$–1, and $1\frac{1}{4}$–$\frac{3}{4}$-in. sizes respectively. The tests show that even reactive low-temperature coke should be burnt on a grate which provides for the admission of air beneath the bars.

Perhaps the most important property of a coke for use in an open grate is its specific reactivity. Whereas in a blast-furnace the need for a readily ignitable coke does not arise, because the coke enters the oxidation zone at a high temperature, and the air-blast is at a temperature higher than the ignition-temperature of the coke, in an open grate ease of ignition of the fuel is an important consideration. Similarly, in a closed stove the reactivity of the coke is not usually of much importance, because a temperature higher than the ignition-temperature of the coke can readily be maintained,

though, as already pointed out, an unreactive coke will not remain alight when the rate of combustion is very low. From an open grate, however, loss of heat to the surroundings is necessarily high, and, if a pleasing fire is to be maintained, each fresh charge of coke

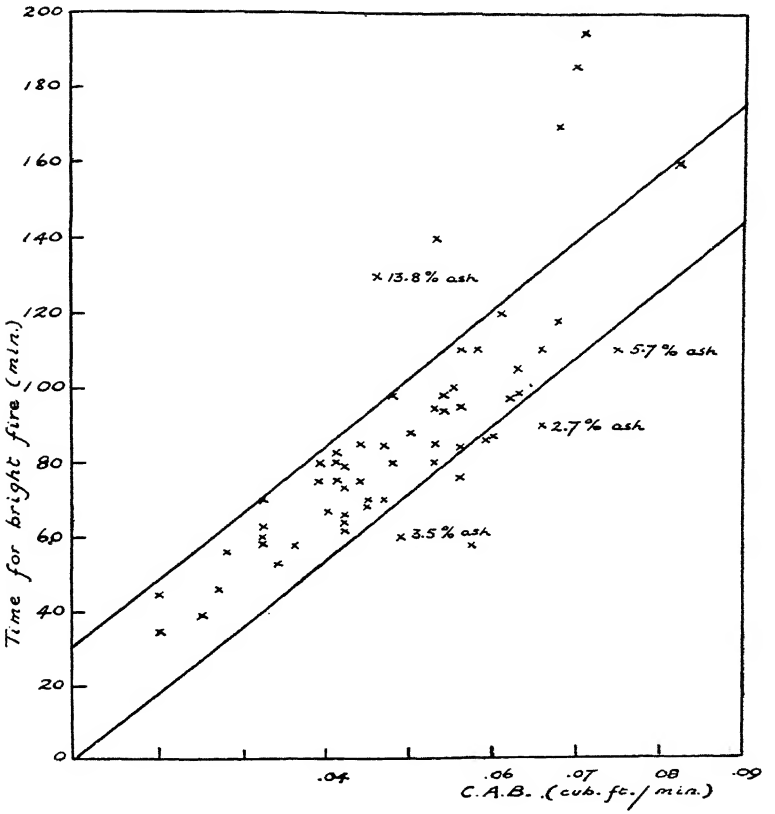


FIG. 43.—The Relationship between the C.A.B. of Coke and the Time for "Bright Fire" in an Open Grate.

must be raised rapidly to its ignition-temperature and active combustion should not be long delayed. The value of a coke for use in open grates can therefore be judged by the time taken for a bright fire to be produced by it.

The time taken by a coke to provide a "bright" fire, *i.e.* a fire radiating sufficiently to warm a room, can be related to its reactivity as measured by its "critical air blast" (C.A.B.). Such

a relationship is indicated in Fig. 43, which records the results of tests made with an "Eagle No. 214" grate using hand-screened cokes of 1½–1-in. square mesh. The grate and the radiometer used for these tests are described in an appendix to this Chapter.

The cokes were built up to a standard level in the grate, the weight taken being usually about 12 lb., but of necessity varying with the bulk-density of the sample. The fire was lighted by gas-jets which were turned out as soon as it was considered that the bed of coke was hot enough for combustion to continue. With low-temperature cokes, the gas could be turned off when a few pieces had begun to glow brightly, but with unreactive cokes the whole of the bottom layer of the bed had to be red-hot.

For reactive low-temperature cokes the time taken to provide a bright fire, under the conditions of test, was 35 to 45 min., but with ordinary by-product oven cokes it was usually more than an hour, and with some of the least reactive cokes it was two hours or more. If a time of one hour be regarded as satisfactory, the C.A.B. for the coke should not exceed 0·045 (see Fig. 43); or, if the time to obtain a bright fire be extended to 80 min., a C.A.B. of not more than 0·055 should suffice. These values are lower than are usual for metallurgical cokes. From Fig. 43 it can also be concluded that, for cokes of the same C.A.B., a bright fire is produced most quickly by those of particularly low ash-content.

TABLE LXIV.—MEAN RATES OF COMBUSTION OF COKES IN OPEN GRATES.

Coke No.	Description	Bulk Density (lb. per cu. ft. of dry coke.*)	C.A.B.	Mean Rate of Combustion (lb. of fixed carbon per hr.) for Different Sizes ‡ (in.)					
				2¼	1¾	1½	1¼	1	¾
	"Coalite" .	19·6	0·020	2·04	2·12	—	2·16		1·80
95	F.R.B. low-temp.	15·4	0·025	2·38	2·21	2·06	2·16	1·94	1·80
	Hearth coke .	23·2	0·020	—	2·42	—	2·35	—	1·98
94	"Carbolux" .	27·7	0·032	—	2·08	—	2·41	—	—
82	Reactive oven coke † . .	24·0	0·041	—	2·21	—	1·93	—	1·88
90Am	Barnsley . .	22·9	0·052	—	1·96	1·97	2·04	—	—
90Ac	Barnsley . .	21·8	0·055	—	2·14	1·93	2·14	1·93	—
93	Durham (low ash)	23·4	0·064	—	1·75	1·86	1·87	1·70	—
33C	Barnsley (c) .	26·1	0·062	—	—	2·10	2·05	1·75	—
92B	Derby (c) . .	25·6	0·072	—	2·01	2·05	1·90	1·82	—
31Ct.	Yorks. Beehive .	—	0·082	—	—	—	2·05	—	—

* Mean values for coke of 2–1 in. size.

† Made from a blend of coking and non-coking coals in 16-in. ovens and coked in 10 hours.

(c) Compressed charge used for coke-making.

‡ The size given is the mean for a half-inch range, e.g. 2¼ is coke of 2½–2 in.

Once a bright fire has been established, all cokes appear to burn at about the same rate, under the same conditions, whatever their reactivity. This is illustrated by the records in Table LXIV of the mean rates of combustion per hour of the "fixed carbon" of a number of cokes (moisture, volatile matter and ash deducted). The combustion periods were calculated over the time when radiation first rose to, and finally fell to, 100 B.Th.U. per sq. ft. per hour during a normal test lasting about 8 hours.

Above about 1-in. the size of the coke does not materially affect the rate of combustion (see Table LXIV), but with sizes 1-in. or less the passage of air through the fuel-bed is hindered. Large coke burns satisfactorily and a bright fire has been obtained with it, but it is not easy to ignite a fuel-bed of large coke in such a way as to obtain a bright fire quickly. In general, it has been found that cokes of 2–1½ in. size with a C.A.B. of over 0·055 cannot be used to start a fire in an open grate unless a considerable quantity of gas is used to ignite them. The range of size of coke that can be successfully used in an open grate is widened if the coke is highly reactive. The time taken for different sizes of cokes to produce a bright fire is illustrated in Fig. 44. The minimum time for most cokes was with 1½–1 in. size, but when the C.A.B. was low (less than 0·04) the time was not affected by the size.

Fig. 45 records for different sizes of coke the hourly radiation per lb. of "fixed carbon" burnt per hour, which affords a measure of the thermal efficiency of the fire. In Fig. 46 maximum radiation during the initial period of the test and after refuelling is recorded for the different sizes of coke, and Fig. 47 records the average temperatures at the top of the fire when there was the maximum radiation during the period after refuelling. The results in general show that, for a wide range of reactivity of the cokes, the best results were obtained with 1¾–1¼ in. sizes.

That the radiation and the temperature should fall with the smaller sizes of cokes is understandable, for their rate of combustion is lower, but the fact that they should tend to fall also when the coke is of larger mean size than 1½-in., whereas the rate of combustion does not fall (see Table LXIV), indicates that the radiation is in part influenced by the amount of burning surface exposed. Apparently there is an optimum amount of surface, and thus an optimum size of coke, for the highest thermal efficiency of the fire.

It is not sufficient, however, to say that coke of a certain size gives the most efficient performance, thermally, in an open grate, for the over-ruling consideration regarding a fire of smokeless fuel is whether or not it is pleasant. A fire of small coke which burns at a slow rate with a low surface-temperature would at once be condemned as an unsatisfactory substitute for a coal fire. A fire of 1½-in. pieces, which often give the best performance with ordinary by-product oven cokes, is liable to be of an unpleasingly uniform appearance, the numerous pieces (about 500 in a full grate) tending

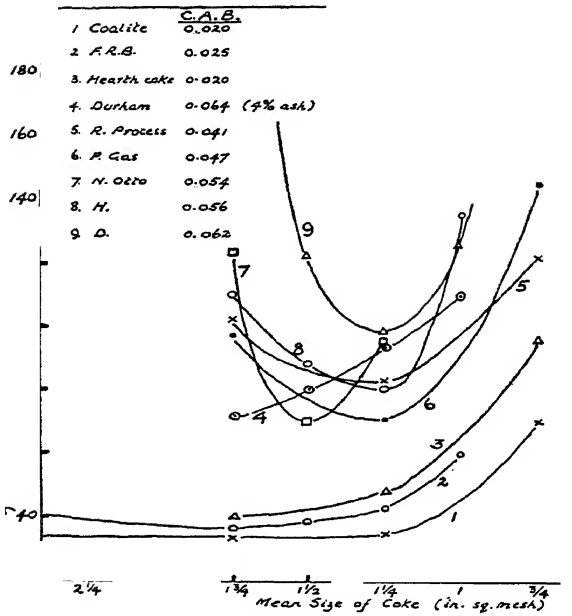


FIG. 44.—The Effect of Size on the Time for "Bright Fire" for Cokes of Different C.A.B.

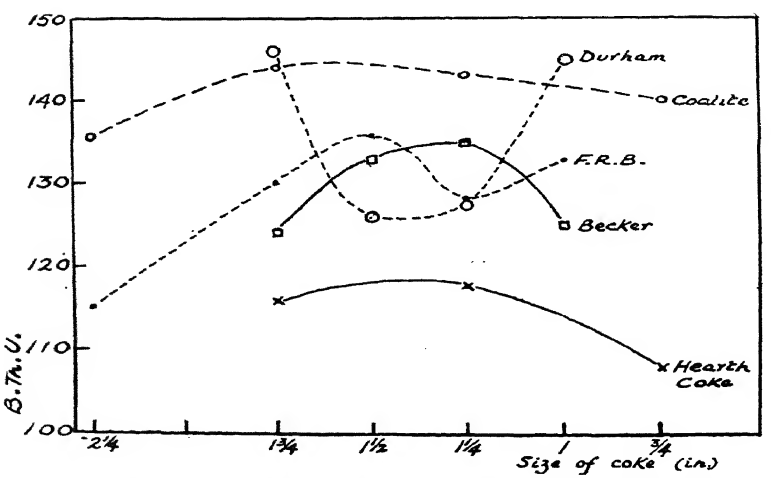


FIG. 45.—The Effect of Size of Coke on the Mean Hourly Radiation per lb. of Fixed Carbon.

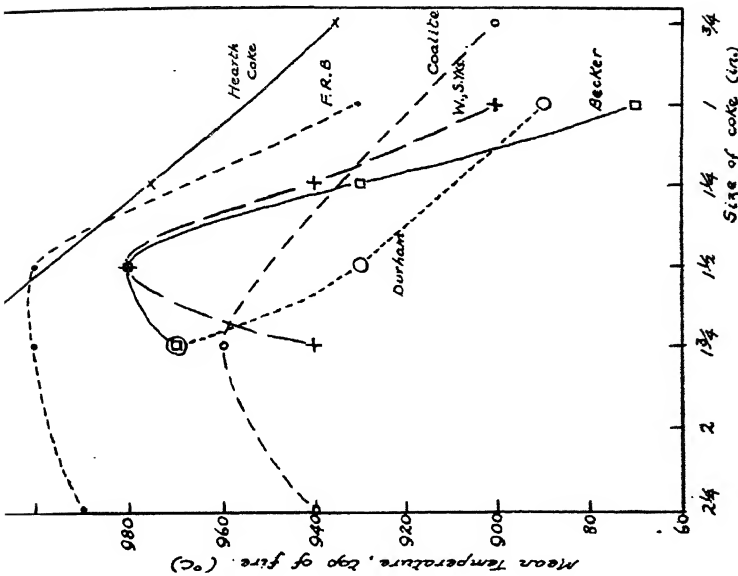


FIG. 47.—The Effect of Size of Coke on the Mean Temperature of the Surface of Fires in an Open Grate.

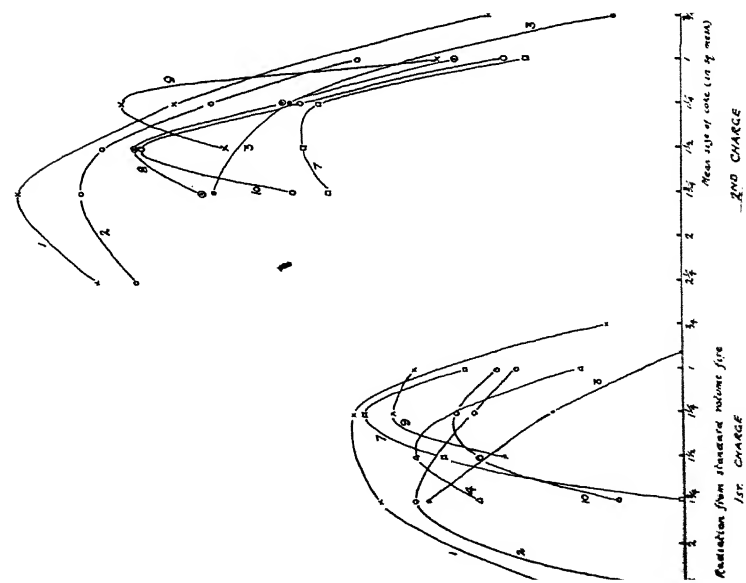


FIG. 48.—The Effect of Size of Coke on the Maximum Radiation in an Open Grate (Cokes listed in Fig. 44)

to cause the radiation from the heart of the fire to be masked. When the size of the coke is increased to $1\frac{3}{4}$–$1\frac{1}{4}$-in. the number of pieces in a full grate drops to about 300, and with coke of 2–$1\frac{1}{2}$-in. size it drops to about 200. With these larger sizes, the masking of the radiation from the heart of the fire is not so pronounced and glimpses can be obtained of surfaces at a bright yellow heat. With still larger sizes of coke the surface of the fire will present black uninflamed patches, and this non-uniformity of the surface is quite attractive. Such a fire can be maintained after the brickwork of the grate has become thoroughly heated, but, as already stated, the use of pieces of coke larger than 2-in. in size would delay the production of a bright fire at the outset.

A coke of low bulk-density diminishes in volume so rapidly as it burns that, as soon as each fresh charge develops the maximum radiation, the fire begins to shrink visibly. The bulk-densities of the 2–1-in. sizes of cokes in Table LXIV vary between 15·4 and 27·7 lb. per cu. ft., and although this has little or no effect on the rate of combustion per pound of the "fixed carbon," it naturally influences to some extent the time taken to produce a bright fire. Lloyd and Hodsman (*Communication No.* 80, 1933, *Inst. of Gas Engineers*) have concluded that the radiating efficiency of a coke depends to a considerable degree on its bulk-density, their conditions of test including the use of a standard size of coke (2–1 in.) and refuelling every hour. The large size of coke used in their tests would have been prejudicial to the performance of the less reactive cokes, and our own tests, in which about 1 cu. ft. of coke was burnt during 6 to 10 hr. in two charges only, do not bear out the suggestion that cokes of low bulk-density have high radiating efficiency. For example, coke No. 95 (Table LXIV), with a bulk-density of 15·4 lb. per cu. ft., gave only 130 B.Th.U. per sq. ft. per hr. per lb. of "fixed carbon" burnt, as compared with 147 B.Th.U. with coke No. 94 of bulk-density 27·7 lb. per cu. ft. A dense coke is of such advantage in keeping low the number of refuelling periods required, and a fire of very light coke can be so displeasing in appearance, that we must regard a high bulk-density as a desideratum, provided that the time taken to get the fire burning brightly is not too long.

The attractiveness of a fire is increased by the presence of flame. With unreactive cokes the flame is small in amount and only apparent at the peak periods of radiation. With reactive cokes, whether they contain little (2 per cent.) or much (10 per cent.) volatile matter, flame is produced through the reduction of carbon dioxide within the fuel bed. Such a production of flame reduces the surface-temperature of the fire. This is in itself an advantage (for a clear coke fire can become unbearably hot) and, although it may reduce the radiating efficiency, adds to the pleasantness of the fire.

The amount and the bulk of ash yielded by a fuel affects considerably its performance, and its appearance, in an open grate.

In a fuel made from coal which has been finely crushed, the mineral matter is uniformly distributed and is, in consequence, liable to form a coating over each lump of fuel as it burns away, thereby reducing its radiating power. A fuel containing 9 per cent. of mineral matter yields not only three times the weight of ash given by a good house coal, but much more than three times the bulk. This ash may be of objectionable appearance after 10 or 12 lb. of fuel have been burnt away, and, as we have already seen, its presence interferes with the burning of the fire. The cleaning of coal to a considerably lower dirt-content than is usual in by-product coking practice is necessary to provide a carbonised fuel for open grates that can successfully compete in favour with coal.

To sum up, we find that the "reactivity" of a coke is the most important property affecting its use in an open grate; that a satisfactory measure of the reactivity can be obtained from the "critical air blast" (C.A.B.); that a C.A.B. not greater than 0·045 is desirable if a bright fire is to be produced within an hour of lighting; that the optimum mean size of the coke pieces is $1\frac{3}{4}$-in. (within a range of 2–$1\frac{1}{2}$-in.); and that the ash-content should be low and of high bulk-density.

APPENDICES

I. THE DETERMINATION OF THE "CRITICAL AIR BLAST" (C.A.B.)

The measurement of the "critical air blast" as a means of judging the reactivity of a coke is due to Blayden, Noble and Riley (*J. Inst. Fuel*, 1934, **7**, 139). The apparatus used, a registered design, is illustrated in Fig. 48. A dried sample of the coke to be tested is sieved between 10 and 20 mesh (I.M.M. standard) and filled up to an etched line, into a container, A, of 1·50 in. (38 mm.) internal diameter, narrowed at a distance of about 1-in. from the top to provide a ledge on which a Gooch disc, forming a bed for the coke, can rest. The narrowed portion of the container is continued over 4 in. and a sleeve is fused on near its lower end. An electric heater of standard design, with three coils fixed in a Sindanyo frame, rests on the top of the container and is surmounted by a Gooch disc. Dry air is passed downwards through the column of coke at a rate of 0·150 cu. ft. per min. (at a temperature of 60° F. and a pressure of 30-in. mercury), whilst an electric current of 124 watts is passed through the heater during five minutes. Under this treatment the coke ignites and the electric current is then switched off and the air-supply reduced to, say, 0·060 cu. ft. per min. If, under these conditions, the coke ceases to burn within, say, 10 minutes, the apparatus is allowed to cool and the test is repeated with an air-supply, after ignition has been effected in the standard manner, of 0·062 cu. ft. per min. Alternatively, if with the air-supply first used, say 0·060 cu. ft. per min., combustion of the coke readily continues during 20 minutes, the test is repeated with an air-supply of 0·058 cu. ft. per min. In this way, by trial and error, the critical air blast required to maintain combustion of the coke during 20 minutes is determined. The test is based on the finding, illustrated by Fig. 49, that with a certain critical air blast the percentage loss of weight of a coke by combustion rises sharply, and that with an increase over this critical air blast of not more than 0·01 cu. ft. of air per min. at least 50 per cent. of the coke-sample will be burnt in 20 minutes.

We use a simplified container (see Fig. 50) consisting of a plain tube of fused

silica, 8 in. long and of 1·42 (36 mm.) * internal diameter. Support for the coke-sample is provided by a disc of copper gauze resting on four legs fused within the tube not less than 1-in. from the top.

It has been found that the "standard" electric heaters vary as regards the distance between the coils and the surface supporting them, and five minutes may

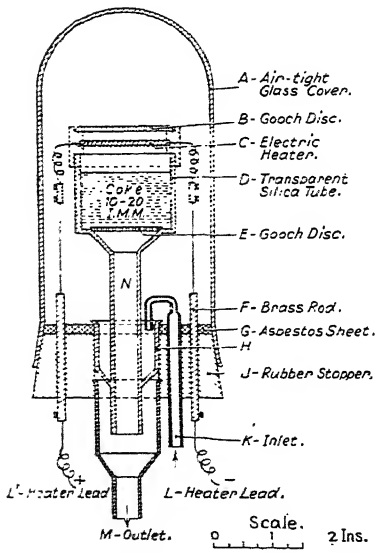


FIG. 48.—Apparatus for Determining Critical Air Blast (C.A.B.). Northern Coke Research Committee.

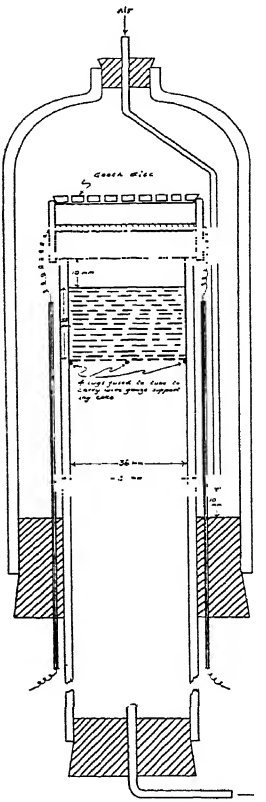


FIG. 50.—Apparatus for Determining Critical Air Blast. Midland Coke Research Committee.

be found insufficient to cause uniform ignition of the coke-samples. With an unreactive coke, there may be merely a bright spot at the centre of the surface; with one of medium reactivity, the ignited area may spread to the wall of the tube; and with a highly-reactive coke the ignited zone may be of appreciable depth.

* It has been found that the diameter of the tube influences the C.A.B. It has been agreed that a tube of 1·50 in. (38 mm.) internal diameter should be taken as standard and all the C.A.B.'s reported in this book should be multiplied by the factor 1·055. For further description of the test see "The Critical Air Blast Test and its Significance," by W. Brewin and J. K. Thompson, *Fuel in Science and Practice*, 1937, 16, 359.

With such differences in the intensity of ignition, comparison between one coke and another may not be satisfactory, for the beds of coke will be heated throughout to different temperatures before the determinations of " critical air blast " are begun.

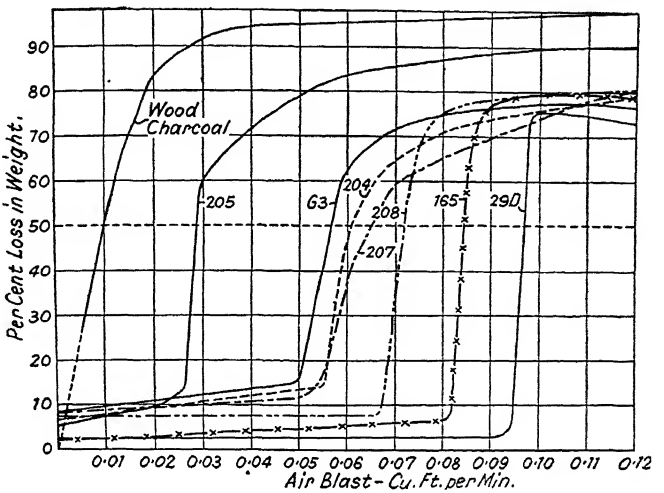


FIG. 49.—Accelerated Combustion of Fuels at the Critical Air Blast (N.C.R.C.)

On the other hand, we have found that, after the zone of ignition has spread to the wall of the tube, the C.A.B. is almost uninfluenced by the duration of the period of ignition, as the following results show :

Duration of Ignition Period (min.)	Condition of Heated Surface	C.A.B. (cu. ft. per min.)
8	Small glowing area	0·150
8½	About half area glowing	0·094
9	Whole area glowing	0·074
9½	Whole area glowing	0·073
10½	Whole area glowing	0·071

These results suggested that the adoption of a " standard intensity " of ignition in place of a " standard time " would be more likely to enable comparable results to be obtained by different experimenters. We therefore decided to regard the ignition of the whole of the surface of the sample of coke as representing a standard intensity of ignition. For ignition, we use a current of 1·8 amp. at 100 volts in the heater.

With this method of test we have found that determinations of the C.A.B. of a series of blends of a reactive with an unreactive coke yield a smooth curve. With the critical air blast, it may be difficult to observe whether combustion is maintained during 20 minutes, and a useful check is obtained by allowing the exit gases to impinge on the flame of a bunsen burner ; if the flame is tinged green (an effect due to the copper surface supporting the coke), combustion continues.

Usually four or five tests, each lasting about 35 minutes, are required to establish the C.A.B. for an unspecified coke. The values range between 0·010–0·020 for low-temperature cokes and between 0·060–0·070 for by-product oven cokes.

II. TEST COKE GRATE AND WATER FLOW RADIOMETER

The grate used for testing most of the different cokes was an Eagle No. 214, described as a "portable 16-in." It is illustrated in Fig. 51. The grate stands away from the wall, and its external heat-loss is greater than that of a set-in grate. The bottom is 14 in. wide by 7 in. The bars, sloping towards the back at an angle of 15° to the horizontal, are $1\frac{5}{8}$ in. thick and are spaced $\frac{11}{16}$ in. apart; the upper surfaces are serrated to guard against over-heating. The ash-pan is 3 in. deep and has a $4\frac{1}{2}$-in. front plate with six 2 in. by $\frac{1}{2}$ in. air-inlet holes. The cross-section of the grate is rectangular, and the throat opening under the fixed canopy is 4-in. wide. The canopy is 12 in. above the uppermost of three front bars, spaced $1\frac{1}{4}$ in. apart, provided to support the bed of coke. The depth of the bed of coke at the bars is about 4 in. and at the back of the fire it is about 9 in. The curvature of the bars allows of a maximum distance from front to back of the fire of $7\frac{1}{2}$ in.

The radiometer we have used is a modification of the "Richmond" (*J. Gas Lighting*, 1915, **129**, 784) and is similar in most respects to that used at the Fuel Research Station (*Fuel Research Tech. Paper No.* 46). Fig. 51 shows the design and Fig. 52 is a photograph of the apparatus in use. Briefly, the instrument consists of a surface of blackened copper, 6 in. square, to absorb radiated heat; behind this surface is a narrow chamber, containing baffles, within which heat is rapidly transferred to a stream of water; thermometers for measuring the temperature of the water before and after it enters the chamber; and a surrounding water-jacket, through which the heated water from the chamber flows, protecting all sides of the radiometer, except that facing the fire, from loss or gain of heat.

By supplying water at a constant head from an overhead tank and by using an adjustable constant-level control, with a trap to remove air-bubbles, a stream of water can be maintained through the radiometer at a constant rate. The radiometer is fixed 3-ft. from the front bars of the grate and in the position 0 0°, that is to say, focussed on the mid-point of the level of the coke-bed above the bars (0° latitude) and in the centre of the width of the fire (0° longitude). The heat radiated is multiplied by 4 to give the heat per sq. ft. of surface in the standard position, and is reported as an hourly rate in conformity with usual engineering practice. The Fuel Research Board report their results in B.Th.U. per 0·25 sq. ft. per min., and the Scottish Coke Research Committee record theirs in B.Th.U. per sq. ft. per hr. at a distance of 51 in. Our "bright fire" corresponds to 200 B.Th.U. radiated per sq. ft. per hr., which is equivalent to 0·843 B.Th.U. per 0·25 sq. ft. per min. and can be compared with the Fuel Research Board's index of an "intense fire," namely, 1·05 B.Th.U. per 0·25 sq. ft. per min. The Scottish Coke Research Committee regard a radiation of 100 B.Th.U. per sq. ft. per hr. at a distance of 51 in. as the index of a "fairly good fire." This is equivalent to 200 B.Th.U. per sq. ft. per hr. at a distance of 3 ft., i.e. our index of a "bright fire."

III. THE WET OXIDATION OF COKE.

The apparatus used for measurements of the wet oxidation of coke is illustrated in Fig. 53. The method involves measuring the rate of evolution of carbon dioxide from a graded sample of coke when it is oxidised at 100° C. by a mixture of chromic and sulphuric acids. One gramme of the dried coke, sieved between 60 and 100 mesh (I.M.M. standard), is introduced into the reaction-vessel, B, which is $1\frac{1}{4}$ in. in diameter by $7\frac{1}{2}$ in. long, and 50 c.c. of a solution of 20 grammes of A.R. potassium dichromate in 100 c.c. of syrupy phosphoric acid (*d.* 1·75) are added. The reaction-vessel is connected to the purifying and absorption trains and heated in a bath of boiling water whilst a stream of air, free from carbon dioxide, is drawn through it at a rate of 50 c.c. per minute. The weight of carbon dioxide evolved during $2\frac{1}{2}$-hr. is determined by weighing either of the absorption-tubes, F. or G. This weight, in milligrams, is recorded as the index of the test.

The values obtained are, for low-temperature cokes, about 100, for Yorkshire blast-furnace cokes, about 150, for Durham foundry cokes, about 200. Graphite has given a value of 824, and other values have been, electrode carbon, 1044, retort carbon, 258 to 293, pitch coke, 284, diamond and sugar charcoal, about 42.

IV. FAST-DRIVING COKE FOR BLAST-FURNACES.

We discussed in our first report the views of Bell, Howland and Koppers regarding the character of a coke which would give, in a blast-furnace, a low consumption per ton of iron. In the preceding chapter, we have shown that the rate of combustion of coke is dependent essentially on the rate of supply of air to it, and that different

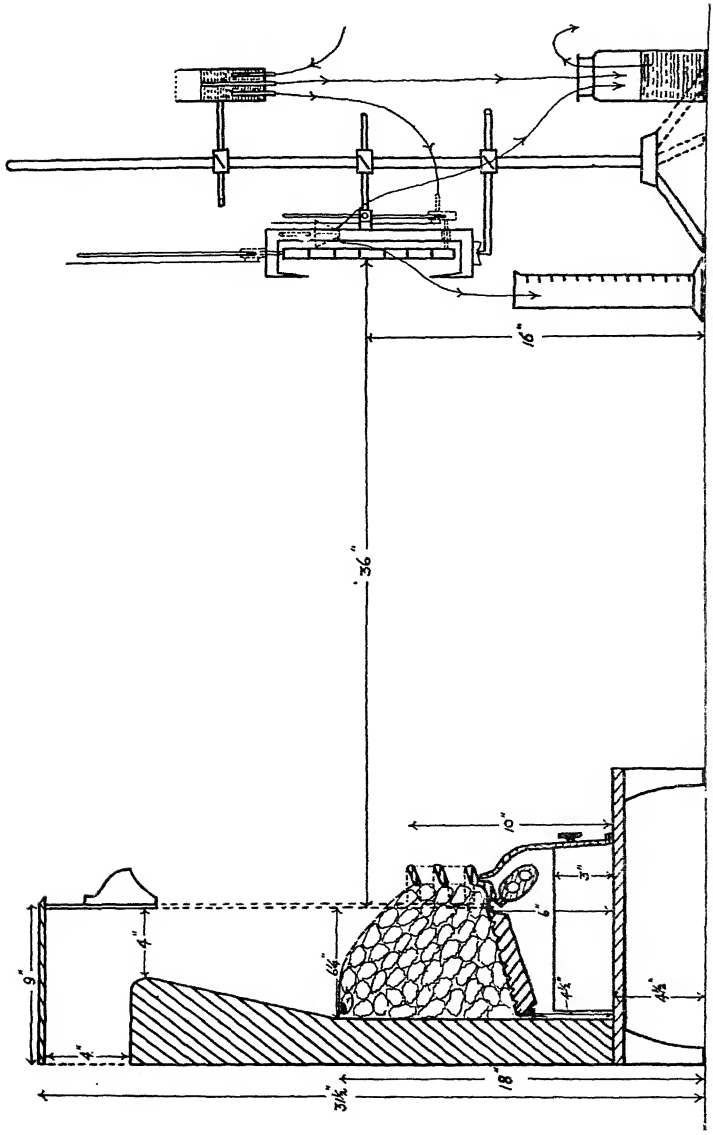


FIG. 51.—Arrangement for Determining Radiation from a Coke Fire. Eagle Grate No. 214 and Radiometer.

cokes of the same size differ but little in their specific reactivity with oxygen. Moreover, cokes of low specific reactivity give the highest maximum temperatures of combustion. Although, under the conditions of blast-furnace practice in Great Britain during the first quarter of the present century, cokes of low specific reactivity, such as Durham cokes, have gained a good reputation, we suggest that the present trend of blast-furnace practice towards faster rates of drive may tend to make "unreactive" cokes less desirable. The reason for this, we suggest, is that the faster the rate of drive the more likely is incompletely-reduced ore to be found in the

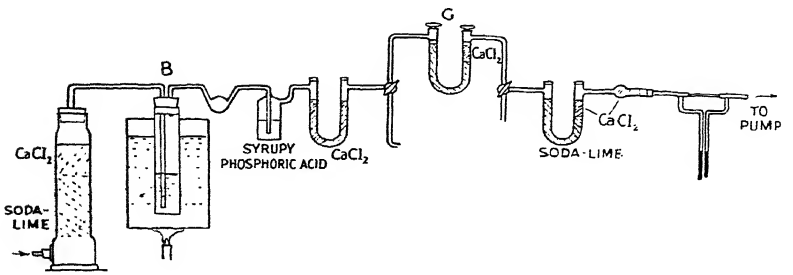


FIG. 53.—Apparatus for Determining the Wet Oxidation of Coke (N.C.R.C.).

reducing zone of high temperature immediately above the tuyeres; whilst for the final reduction of such ore, as well as for the reduction of oxides of silicon, phosphorus and manganese to the metallic state, solid carbon is the reducing medium and, for this purpose, should be "reactive." This view of "reactivity," it should be noted is different from that previously considered.

It is only necessary for a proportion of the coke to be reactive, for Evans, Reeve and Vernon (*Journ. I.S. Inst.*, 1931, **1**, 98) found that the carbon consumed other than at the tuyeres is most commonly 350 lb. per ton of pig, or about 15 per cent. of the total. In German blast-furnaces the carbon consumed other than at the tuyeres amounted most commonly to 20 per cent. of the total (Eichenberg, quoted by Evans, Reeve and Bailey, *loc. cit.*, p. 97). The amount of this carbon may vary from under 50 to over 700 lb. per ton of pig. It seems reasonable to assume that with fast rates of drive there is a need for a reactive solid carbon for the reduction of metallic oxides and for carburisation, so that with very fast rates of drive "unreactive" cokes may not be desirable. Although unreactive cokes are usually of high impact hardness, and thus facilitate fast rates of drive, it may be that the inclusion of about 20 per cent. of a more reactive coke, despite its greater liability to produce breeze, would be beneficial.

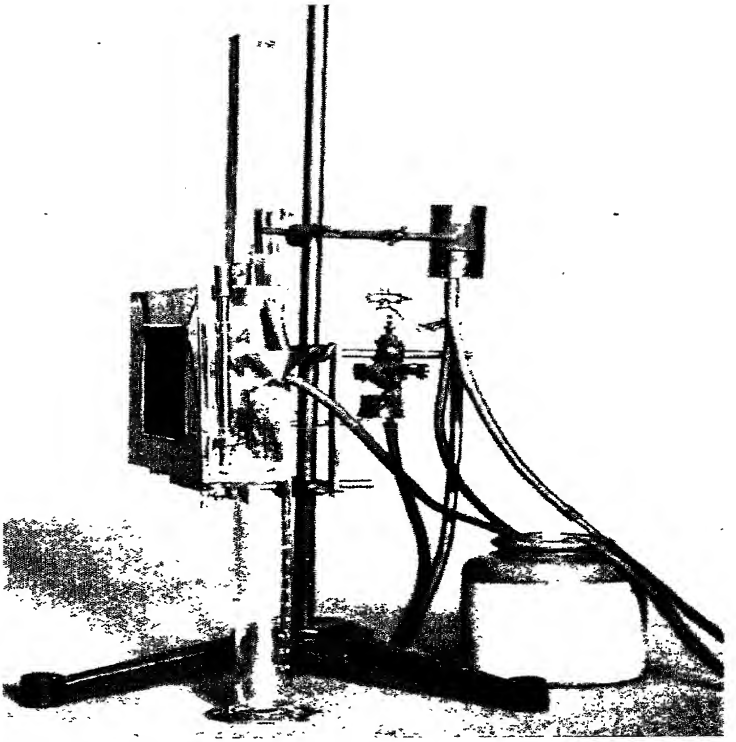


FIG. 52.—View of Radiometer.

CHAPTER XII

THE SPECIFICATION OF COKE

INTRODUCTION

A RIGID specification for metallurgical coke is not possible, for, although its possession of certain qualities is always necessary, fairly wide variation in those qualities is permissible, in blast-furnace coke, for example, dependent on such matters as the type of iron made, the volume of slag per ton of iron and the rate of drive of the furnace. It is of interest to consider this statement in some detail.

The coke required when pig iron is being made for the acid steel process must have a low phosphorus-content, for phosphorus is not removed during that process, but when the pig iron is required for the basic steel process, or for forge and foundry work, the phosphorus-content of the coke is of no moment.

When pig iron is being made from rich ores containing, say, 40 to 60 per cent. of iron, the volume of slag per ton of iron is less than when lean ores, containing about 20 to 30 per cent. of iron, are used. Since the solubility of sulphur in the slag is limited, the amount of sulphur that can be eliminated from the raw materials fed to the blast-furnace depends largely upon the volume of slag removed per ton of pig iron made. With rich ores and a coke of low ash-content, the amount of slag formed is small and it may therefore be desirable to specify a limit to the amount of sulphur in the coke. With lean ores, such as those of Lincolnshire and Northamptonshire, the volume of slag is sufficient to remove up to, say, $2\frac{1}{2}$ per cent. of sulphur in the coke and, in practice, the sulphur-content of the coke need not be specified.

The amount of ash that can be tolerated in blast-furnace coke depends upon the richness of the ore smelted. In India ores containing up to 50 per cent. of iron are commonly used, and the coke may contain 15 per cent. or more of ash without detriment; a fortunate circumstance, since the inherent ash of Indian coking coals is high and normal methods of coal-cleaning do not remove much of it. Similarly, in New South Wales, when ores containing over 50 per cent. of iron are smelted, the presence of up to 15 or 16 per cent. of ash in the coke does not adversely affect the working of the furnaces. When lean ores are used, however, the less the amount of ash in the coke the better, and a lower percentage than is now usual in cokes in the Midland area (about

9 or 9½ per cent.) should be particularly advantageous when a rapid rate of drive of the furnace is desired.

The Rate of Drive of Blast-Furnaces.—The rate at which any blast-furnace can be driven depends not only on the type of ore available but on the type of pig-iron desired. The rate of drive of blast-furnaces has increased considerably since coke first began to be used, and it may be that the extent to which the qualities desirable in coke are dependent on the rate of drive of the furnace has been generally over-looked.

When the use of coke in blast-furnaces became general in the Coalbrookdale area of Shropshire, about the year 1760, the furnaces were not much more than 30 ft. high and the output per furnace was about 20 tons of pig iron per week. By 1790, when coke blast-furnaces became common in other areas, their height had been increased to about 40 ft. and their output to about 40 tons per week, a development due to the use of steam-driven cylinder blowers; and during the first decade of the nineteenth century the height was increased to 50–70 ft. (the taller furnaces being in South Wales) and the output to 50 tons per week. Up to this time the coke used was "hearth" coke made from lump coal, carbonised at a low temperature under a layer of coke dust. Such coke is reactive. The use of hot blast enabled wider hearths to be built in furnaces of greater capacity, and by the time the Cleveland iron industry began (about 1850) outputs of 200 to 220 tons per week from furnaces 45 to 50 ft. high were common in Staffordshire and in Scotland, where reactive cokes made from coals of low rank were used. With the unreactive beehive cokes of Durham, it was found that an increase in the height of a blast furnace to 80 ft. caused a reduction in coke consumption from 35 to 22 cwt. per ton of pig iron, with an increase in production to 500 tons per week.

The progressive increase in average capacity of blast-furnaces in Great Britain during the past 60 years is shown in Fig. 54, which gives the records over the periods 1875 to 1922, 1922 to 1931 and 1931 to 1937. Over the period 1875 to 1910 there was an increase from an average of 200 tons to one of nearly 600 tons per week, and from 1922 to 1931 the average production rose from 700 to nearly 1000 tons per week. A new trend towards furnaces of greater capacity then set in, whereby the average weekly production was raised to 1250 tons in 1935 and to 1340 tons during 1936, individual furnaces of 1500 to 2000 tons weekly capacity operating on the lean ores of Lincolnshire and Northamptonshire, and of as much as 4000 tons capacity on rich hæmatite ores. The faster driving has been gained by the use of furnaces of larger cubic content, for, in general, the time of descent of the stock bears no direct relationship to the rate of production of iron (*see* E. C. Evans, L. Reeve and M. A. Vernon, "Blast furnace data and their correlation, Part II," *J. Iron and Steel Inst.*, 1931, *Appendices IA and IB*).

For a given furnace, however, an increase in the rate of drive means an increase in the rate of descent of the stock.

Records show that for furnaces of capacity ranging between 900 and (in foreign countries) 7000 tons per week, the time of descent

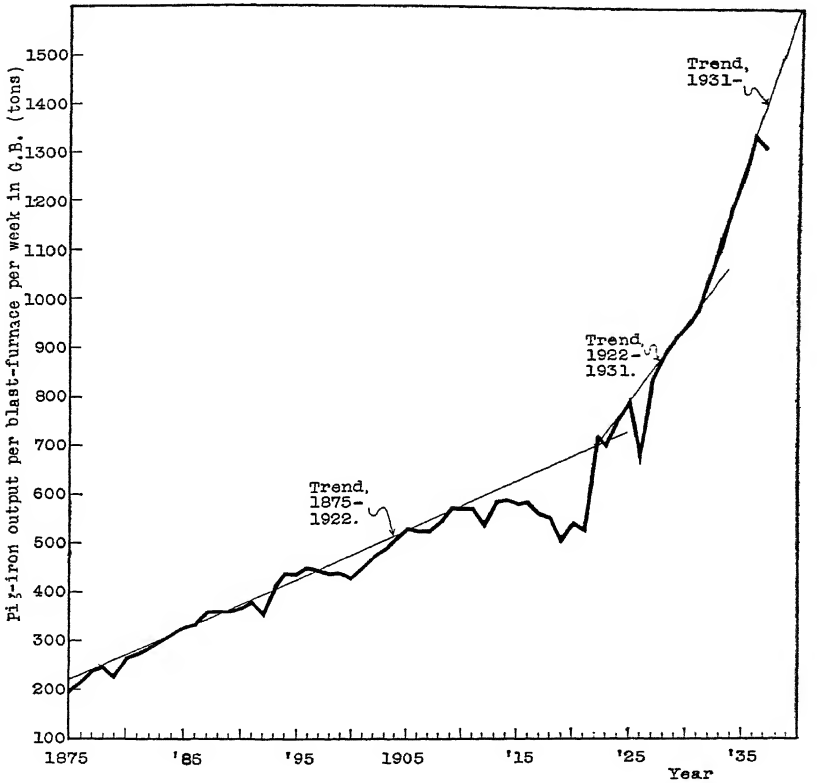


FIG. 54.—Average Pig Iron Production per Furnace for Great Britain, 1875-1937.

of the stock may be under 9 hours. The usual time is from 10 to 15 hours and, as a general rule, if the time exceeds 20 hours the capacity of the furnace is low. It is probable that, since the time (1875 to 1895) when Sir Lowthian Bell was studying the qualities to be desired in blast-furnace cokes, the average time of descent of the stock has been halved or, in the few furnaces of large capacity (over 3000 tons of iron per week) in this country, reduced to one-third. As a consequence, it may be that a coke of low reactivity, such as beehive coke, has ceased to be desirable; for the reason

that the more rapid descent of the stock demands a coke that will react rapidly, as a solid, with solid oxides above the hearth zone (see Appendix IV, Chapter XI).

The Quality of Coke and the Rate of Drive of Blast-Furnaces.—For a particular furnace, an increase in the rate of drive involves an increased rate of passage of air into the furnace. A limit to the rate at which a furnace can be blown is set by the physical quality of the coke, which should be of high impact hardness so that it will not produce much breeze during the severe handling that is almost inevitable in blast-furnace practice, and of high surface hardness (*i.e.* of low "abradability") so that it will not produce much dust during its passage down the shaft. If the coke charged to a blast-furnace contains much breeze, or if much dust is formed from it by abrasion, the air-blast and the reducing gases formed near the tuyeres cannot pass uniformly through the charge, the contact between solids and gases is thus unsatisfactory, and a longer time may be required to complete the reactions in the shaft. With the tendency that there is to increase the rate of drive of blast-furnaces the physical quality of the coke has become of increased importance.

In the past, the quality of blast-furnace coke has not been commercially defined. In times of restricted trade the poorer grades of coke have found little demand, and there has been, in consequence, a general tendency to improve the quality. When trade has been brisk, the demand for coke has embraced the poorest grades, and the quality of the better grades has not always been maintained. These tendencies are illustrated in Fig. 55, in which the average consumption of coke per ton of pig iron made in Great Britain over a period of years is plotted against the price of blast-furnace coke delivered at Middlesbrough. It will be seen that the peaks on the graphs for coke-consumption and the price of coke correspond. In times of good trade, the quality of the coke, in high demand, has deteriorated, and it has been necessary to use more of it per ton of pig iron* (*see* R. A. Mott, "The commercial evaluation of coke," *J. Inst. Fuel*, 1931, **5**, 193).

Before the year 1913 less than 50 per cent. of the metallurgical coke produced in this country was made in by-product ovens, and the marked increase in coke-consumption and the reduction in the rate of drive of furnaces in the earlier years of peak demand can be related in part to the difficulty of "forcing" beehive ovens without detriment to the coke. Since 1913, the production of metallurgical coke in by-product ovens had risen from 56 to 75 per cent. of the total in 1918, and to 95 per cent. in 1928, and since 1928 many new plants, with modern arrangements for quenching and screening the coke, have been erected. Moreover,

* It should be noted that the consumption of coke has been influenced to some extent by the amount of ore used per ton of pig iron.

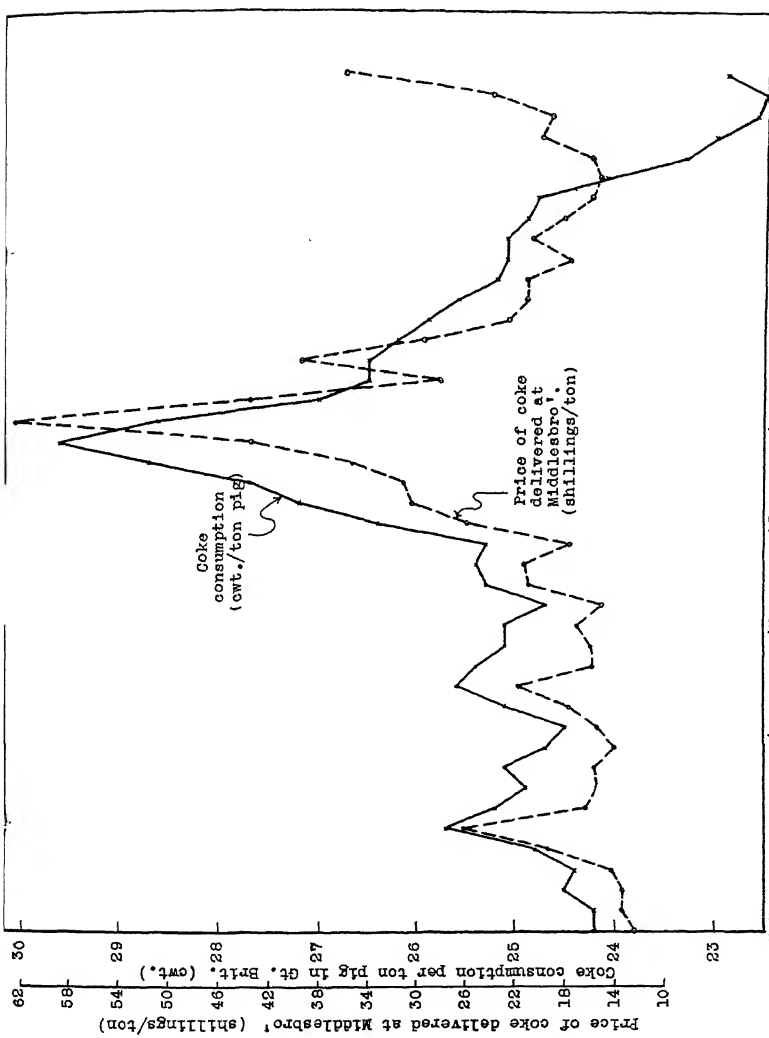


FIG. 55.—Graph Illustrating Deterioration of Quality of Coke (with increased Consumption per ton of Pig) in Periods of Great Demand (and High Prices).

coal-cleaning has made considerable progress since 1913, when only about 14 per cent. of the saleable coal was so treated, the amount cleaned in 1937 being 43·8 per cent. There should therefore no longer be much difficulty in maintaining the quality of coke during periods of heavy demand, provided that suitable coking slacks are available.

Actually, the provisions of the Coal Mines Act, 1930, have often rendered it difficult for a coke-making plant to be supplied with the coals desired. In the Midland area, the increase in the demand for coke since March, 1934, which accelerated during the winter of 1935-36, has made it necessary to supplement the usual coking slacks by slacks of inferior coking properties, or to use the nut coals from the good coking seams, which do not yield as hard cokes as the slacks. This shortage of the most suitable coking slacks no doubt accounts for the recent deterioration in quality manifest in the Midland cokes, as exemplified by the following averages for about 20 different cokes :

	1932	1933	1934	1935	1936	1937
Mean moisture-content .	3·5	3·7	3·5	3·5	4·0	3·9
Mean ash-content . .	9·0	8·8	8·8	9·0	9·4	9·5
Mean shatter index (1½-in.)	78·5	79·4	79·2	79·5	78·3	78·2
Coke production in Midlands* . . . (million tons)	3·69	3·61	4·57	4·82	5·21	5·63

* Excluding Lancashire and Staffordshire.

It should be observed that during 1937 the demand for blast-furnace coke increased by 56 per cent., compared with 1933.

The Average Quality of Midland Cokes

A study of the average quality of Midland cokes during recent years shows that, until the demand became excessive, it was well maintained.

Moisture-content.—Fig. 56 shows the annual mean moisture-contents of cokes delivered from twelve Yorkshire coking plants during the past nine years, and records also the total annual outputs of coke in the Midland area. The total production of coke in the Midlands reached a new record in 1929, and this record was exceeded in 1934 and subsequent years ; nevertheless, for the plants under review the mean moisture-content of the cokes of 1937 was 1 per cent. less than that of the cokes of 1929. The twelve coking-plants under consideration have a capacity about 47 per cent. of the total nominal coke-producing capacity of South Yorkshire. For cokes representing 83 per cent. of the total production the moisture content was less.

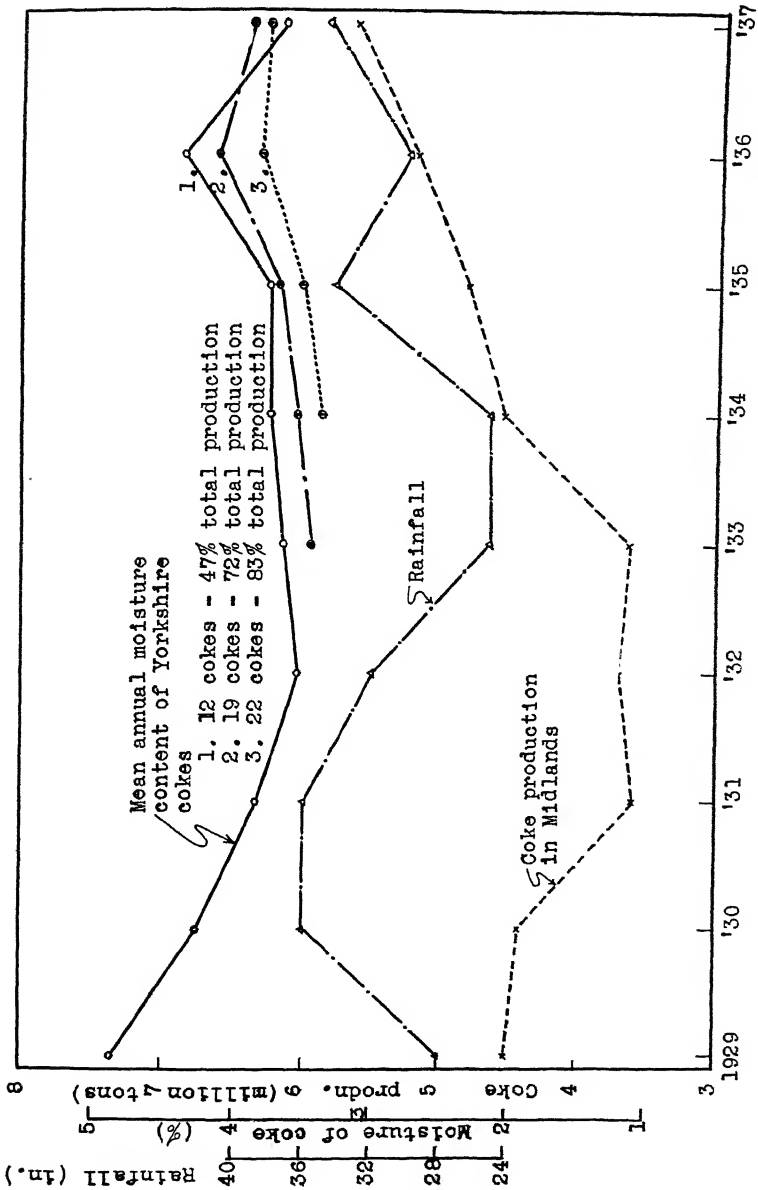


Fig. 50.—Mean Annual Moisture Content of Yorkshire Cokes for Different Years Compared with Rainfall and Total Coke Production

One reason for the reduction in the moisture-content of South Yorkshire coke during recent years, despite the increased output, is indicated in Fig. 57, which compares the quarterly moisture-contents of eleven cokes hand-quenched on an inclined or flat bench with those of eleven cokes quenched at central plants. It will be seen that the hand-quenched cokes contained from 2 to 3 per cent. more moisture than the centrally-quenched, the difference increasing with the greater production of recent years. The adoption of central quenching at the newer coking plants of South Yorkshire has counteracted the tendency towards over-watering that there is with hand-quenching during rapid rates of throughput, when the quenching of a newly-pushed charge whilst a number of charges of coke lie on the bench renders the addition of water to coke already quenched unavoidable.

Fig. 57 shows also that variations in the mean quarterly moisture-contents of coke may be related with the rainfalls of the periods, and an examination of results recorded over a longer time (Fig. 58) shows that the moisture-content of coke tends to be at its highest during either the first or, more frequently, the fourth quarter of the year. During the last five years of Fig. 58 the highest rainfall has been during the winter months. In earlier years, heavy rainfall during the summer months has not materially affected the moisture-content of the coke, presumably because of such factors as sunshine and wind favouring the evaporation of water. The moisture in coke is lost rapidly when it is freely exposed to air and more rapidly in an air-current. Whereas during the summer the water added to coke by heavy rainfall is for the most part lost by evaporation, either from the tops of wagons or as the coke is exposed on the bench, during the winter months evaporation is ineffective. The abnormally heavy rainfall during the fourth quarter of 1935 may be associated with a general increase in the moisture-content of the coke then produced. This increase was less for centrally-quenched than for hand-quenched coke, perhaps because central quenching was adopted mostly at newer plants which also had improved arrangements for screening out small coke, which retains the most moisture, before loading. For many of the cokes the moisture-content has been consistently less than 2·5 per cent., whilst for others, hand-quenched, it has been 5 per cent.

A rainfall of 1 in. is equivalent to the addition of 2·3 per cent. of moisture to 10 tons of coke in a wagon, but all rainfall is not retained by coke. A test wagon of 7 tons of coke exposed during 16 days to 2·2 in. of rain during January (equivalent to 7·3 per cent. of moisture in the coke) increased in moisture-content by only 1 per cent. The increased moisture-contents of cokes in the winter months is therefore only in part due to added moisture from rainfall, and in part due to the lower rate of evaporation when coke is exposed on the bench after quenching. The greater

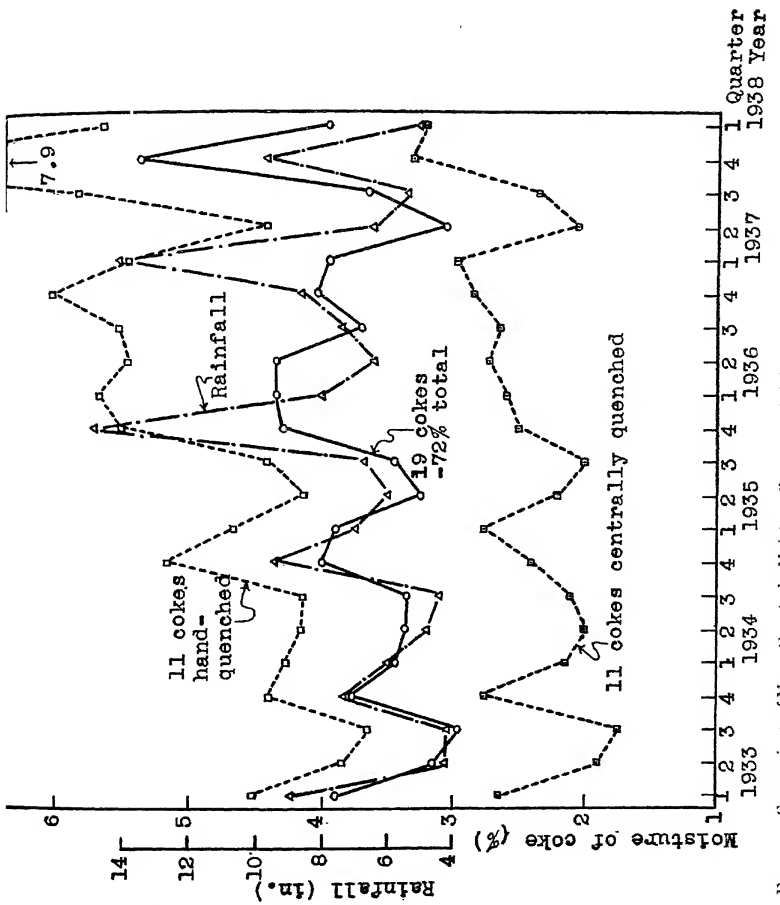


FIG. 57.—Comparison of Mean Quarterly Moisture Contents of Cokes Centrally-Quenched and Hand-Quenched. Comparison of Mean Quarterly Moisture Contents of Yorkshire Cokes and Mean Rainfall.

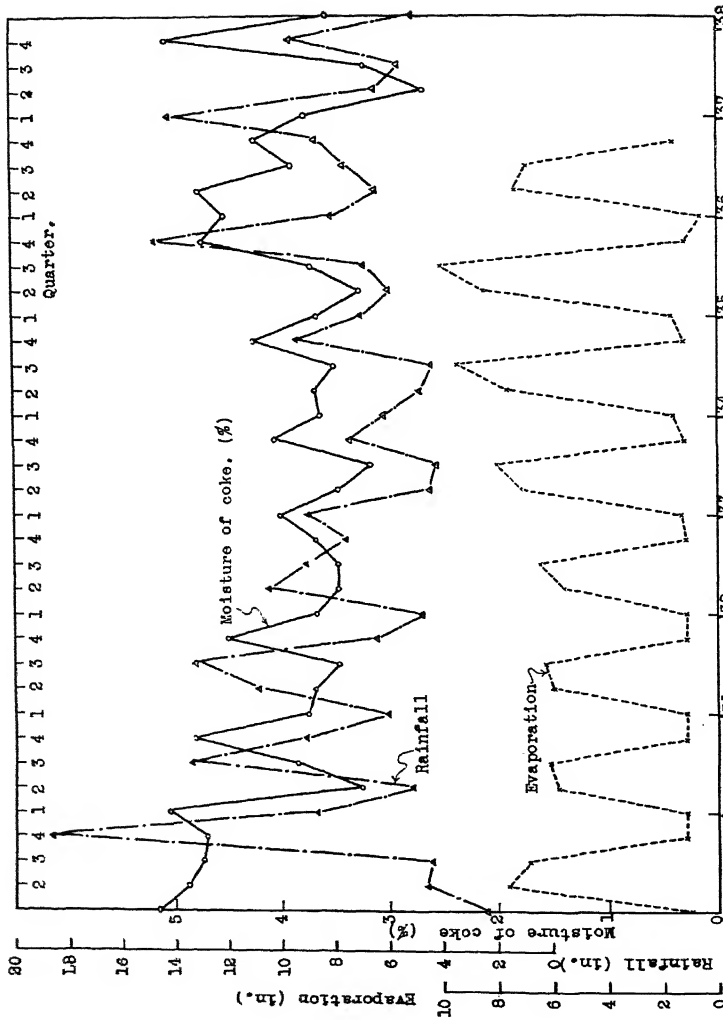


FIG. 58.—The Relationship between Mean Quarterly Moisture Content, Rainfall and Evaporation.

the demand for coke the shorter is the period of exposure on the bench.

Ash-content.—The mean annual ash-contents of a number of Yorkshire cokes delivered at blast-furnace plants during 1931 to 1937 are recorded in Table LXV. The cokes concerned represent from 50 to 75 per cent. of the total production of South Yorkshire.

TABLE LXV.—THE MEAN ASH-CONTENTS OF YORKSHIRE COKES, 1931-1937.

Blast-Furnace Plant	Number of Cokes	Ash-Content of Coke, per cent.						
		1931	1932	1933	1934	1935	1936	1937
A	14	9·1	8·8	—	8·6	—	—	—
B	17	—	9·0	8·8	8·8	9·0	9·4	9·5
Production of coke in the Midlands, million tons . .		3·58	3·69	3·61	4·57	4·82	5·21	5·63

That an expansion of the production of coke by one-third, involving an increased loading of the coal washeries, could be made without increasing the ash-content of the coke, is evidence of the general improvement in the coal-cleaning plants available. During 1935, the mean ash-content of the coke at one plant was 6·6 per cent. and at another it was 12·2 per cent., but with only 8 out of 22 cokes for which records were obtained did it exceed 10 per cent.

Hardness.—The mean 1½-in. shatter indices of a number of Yorkshire cokes during 1931 to 1937 are recorded in Table LXVI.

TABLE LXVI.—THE MEAN 1½-IN. SHATTER INDICES OF YORKSHIRE COKES, 1931-1937.

Number of Cokes	1½-in. Shatter Index						
	1931	1932	1933	1934	1935	1936	1937
17	81·2	—	79·5	78·9	79·0	77·5	77·2
10	—	79·6	80·5	80·4	80·3	79·3	78·5
19	—	—	79·4	79·0	79·1	78·0	77·8
22	—	—	—	79·2	79·4	78·4	78·1
Production of coke in the Midlands, million tons	3·58	3·69	3·61	4·57	4·82	5·21	5·63

The drop in the shatter index after 1931 can be explained by the operation of the Coal Mines Act, 1930, which restricted the production of coal at all collieries and rendered it necessary for coke-oven plants, erected to carbonise their own coals, to be provided with a proportion of less suitable coking slacks, purchased

on the open market. During 1935 the $1\frac{1}{2}$-in. shatter index for 22 different cokes ranged from 71·3 to 86·8, and during 1937 from 71·4 to 87·2 (with one of 61·2).

Future Demands for Coke

The demand for metallurgical coke in the Midlands is likely to increase. The most abundant supplies of native iron ore have been obtained from the Cleveland bed, which has yielded over 335 million tons, but its production has been steadily falling since 1907, when about 6 million tons were mined, and was no more than $1\frac{1}{2}$ million tons in 1934. Alternative, and more extensive, supplies of iron ore exist in Northamptonshire, Lincolnshire and Oxfordshire, and they are more cheaply mined than the Cleveland ironstone. In 1913, 38 per cent. of British pig iron was made on the North-East coast as compared with only 19 per cent. in the Midlands; by 1923, the proportions were about equal (29 per cent. each) in the two districts and so they remained until 1930; by 1935, the Midlands produced 42 per cent.* and the North-East coast 27 per cent. Provided that coke of suitable quality can be made at a reasonable cost, the manufacture of pig iron in the Midlands should continue to supply the major proportion of British requirements.

Table LXVII.—Estimates of Requirements of Coke in the Midlands. (Million Tons)*

Year	Production of Pig Iron	Coke Required for Pig Iron	Total Production of Oven Coke	Coke Available	Deficiency or Surplus,
1927	2·22	3·12	4·55	2·88	−0·24
1928	2·14	3·00	4·62	2·95	−0·05
1929	2·51	3·52	5·02	3·35	−0·17
1930	2·26	3·17	4·76	3·09	−0·08
1931	1·99	2·24	3·89	2·22	−0·02
1932	1·57	2·20	4·03	2·36	+0·16
1933	1·55	2·21	4·01	2·34	+0·13
1934	2·34	3·28	4·96 ‡	3·29	+0·01
1935	2·70	3·78	5·07 ‡	3·40	−0·38
1936	3·18	4·46	5·47 ‡	3·80	−0·66
1937	3·45	4·83	5·90 ‡	4·23	−0·60

* It should be noted that Lancashire requirements are included in Table LXVII, but not in earlier tables, since the iron production in Lancashire and South Yorkshire are recorded together in official statistics.

* Assuming 28 cwt. of coke per ton of pig iron.

‡ Deducting, for 1934, 100,000 tons and, from 1935, 250,000 tons of coke assumed to have been made in Essex.

The demand for pig iron increased suddenly during March, 1934, and has since continued to increase, whilst the consequent demand for coke outstripped production and a shortage resulted.

* During the first quarter of 1936, the percentage was 49, but it fell to 41 per cent. for 1937, owing to the increased demand.

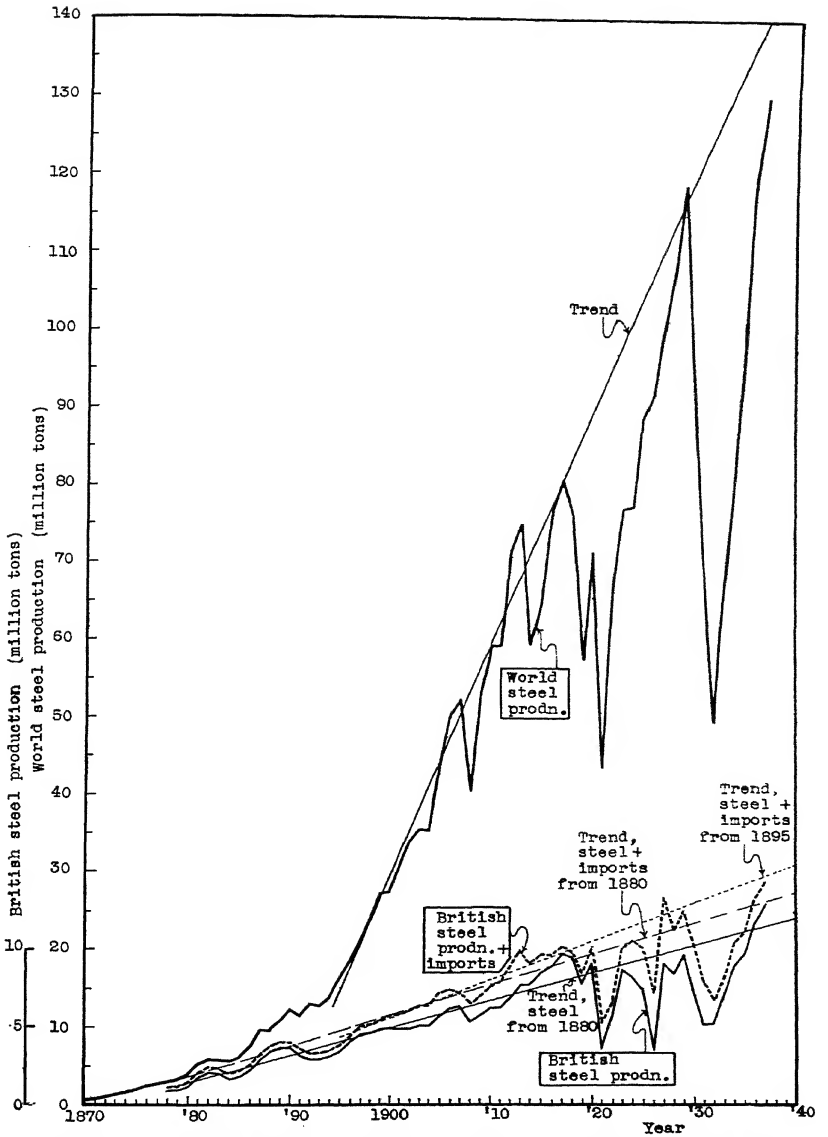


FIG. 59.—Trends of Production of Steel in the World and in Great Britain over Period 1870–1940.

Much of the coke-production in the Midlands (about one-third during the years 1923 to 1930) has been sold for "domestic" purposes, chiefly for central heating, and since this remunerative market has been a reliable stand-by for the coke-makers during the years of depression in the iron and steel industry, it is unlikely that it will be starved. On the assumption that the "domestic" coke market absorbs 1·66 million tons per annum, an estimate is given in Table LXVII of the total requirements of coke in the Midlands from the year 1927. With respect to pig iron production, the "Midlands" include Lincolnshire, Derbyshire, Leicestershire, Nottinghamshire and parts of Lancashire and South Yorkshire; with respect to coke manufacture they include South and West Yorkshire, Derbyshire, Lincolnshire, Lancashire and, from 1934, Northamptonshire.

The deficiency of coke in the Midland area during 1936 and 1937 has been nearly two-thirds of a million tons per annum.

Trade conditions during the post-war years have been so abnormal that it has been customary to regard 1929 as a "boom" year. To obtain an idea of what should be regarded as normal demands for steel, it is necessary to follow the trend of production over a long period. Fig. 59 shows that, from the trend of world-production of steel since 1895, the year 1929 was "normal," whilst for the production of steel in Great Britain the lines of trend suggest that the demand was below "normal" in 1929 (and in 1935) despite the fact that a new high record was set up during that year. The figures for British steel production, however, are influenced by imports, and if the imports are added to the home production the demand for steel is seen to have been above "normal" in 1929. It was $1\frac{3}{4}$ million tons in defect in 1935, but almost normal again in 1937. In 1935, imports of steel were limited by agreement to $\frac{1}{2}$-million tons,* and the "normal" production of steel in Great Britain should be from $12\frac{1}{2}$ to $13\frac{1}{2}$ million tons over the period 1936 to 1940. For such a production of steel, the pig iron requirements (assuming a ratio pig iron to steel of 0·80)† would be from 10 to 11 million tons. The proportion of this pig iron made in the Midlands, taking the mean proportion during the past five years as a basis, would be about $4\frac{1}{2}$ million tons, for which the coke required would be about 6 million tons. Add to this the "domestic" coke and it would appear that, during the next few years, the annual production of coke in the Midlands should be nearly 8 million tons, as compared with about 6 million tons made in 1937.†

* In 1936 and 1937, it was found necessary to abate these arrangements owing to the abnormal demand for steel.

† In the four years ending 1929, the mean ratio of production of pig iron and steel was 0·80; in the four years ending 1937, it was only 0·65, the ratio depending partly on the amount of pig iron available and its price in relation to that of scrap. With a general world activity in steel production the ratio would tend towards the higher figure; with the lower figure the pig iron production would be 8 to 9 million tons, and the proportion made in the Midlands about $3\frac{1}{2}$ million tons, requiring $4\frac{3}{4}$ million tons of coke with a total production of $6\frac{1}{2}$ million tons.

Making due allowance for over-estimation in these calculations, it is evident that, given supplies of suitable coking coals, further expansion of the coke-making industry in the Midlands may be expected if the demands of the iron and steel industry are to be met. The response of the coke-making industry to those demands will naturally depend upon the value placed on the coke.

The Value of Coke

It is not easy to obtain reliable data for the present and anticipated value of coke. Some indication can perhaps be obtained from the recorded values at works in different counties, given in the Board of Trade returns during the years 1905–1920. The figures given are for the total outputs of coke, including gas-coke which amounted to 13 per cent. in South Wales, 5 per cent. in Durham, 25 per cent. in Yorkshire and 17 per cent. in Derbyshire. Averaging the prices given for coke over the period 1905–1920 and assessing the values of Durham coke at 100, the relative values were :

	Mean Price Ratio	Mean Shatter Index Ratio	Actual Mean
South Wales	105	105	
Durham	100	100	
Yorkshire	89	89	
Derbyshire	83		

The records given of the mean $1\frac{1}{2}$-in. shatter indices, based on many tests of representative cokes from the different districts made by the Midland Coke Research Committee, show how closely they correspond with the price ratios, a fact which it is difficult to regard as coincidental.

Coke having a high shatter index (*i.e.* of high impact hardness) enables a rapid rate of drive of a blast-furnace to be maintained. It is therefore important, in view of the modern tendency to increase the rate of drive, that the impact hardness of Midland cokes should be improved. A marked improvement is unlikely in the absence of monetary reward, for, although technically possible, as the work of the Midland Coke Research Committee has shown, the necessary changes in the methods of manufacture entail expenditure. The coke-making industry in the Midlands was developed to deal with the smaller sizes of slack coal, under $\frac{1}{2}$-in. or $\frac{3}{8}$-in., the ovens being built at collieries where such slack would produce good coke. The whole of the slack of the "coking" seams, such as the Parkgate and the Silkstone, together with a proportion of the slack from "steam" coals, such as the Barnsley, often imperfectly blended, is at present used for coke-making. An extension of coke-making activities would demand either the use of the larger sizes of the

coking coals to the extent, perhaps, of coking the whole of the output of a seam, or the careful blending of quantities of steam-coal slacks. The larger sizes of the coking coals produce cokes that are inferior to those obtained from the slacks, and the addition of regulated quantities of fusain or coke dust is necessary to improve the quality.

The Specification of Coke

As an encouragement to the production of coke of high impact hardness, the Midland Coke Research Committee has drawn up a scheme for the sale of coke to specification.

The general principles involved are as follows :—Certain ranges of moisture content, ash content and 1½-in. shatter index are chosen to represent an average or a "standard coke" to which would be assigned a "basic price" by buyers and sellers in the usual way, subject to modification as the ordinary price of coke in this area has been in the past. For qualities of coke better than that of the "standard coke," bonuses would be paid by the coke users, and for qualities below the standard penalties would be exacted. The bonuses and penalties would be percentages of the basic price. Cokes would be assigned to different "classes" with small ranges of moisture, ash and 1½-in. shatter index, and the bonuses or penalties would be judged separately for the three properties specified. The standards applied would be agreed between buyer and seller; the examples here given, however, will probably be found to be applicable to most classes of cokes.

The presence of moisture in coke is detrimental mainly because it reduces the proportion of carbon, and it increases the cost of transport. The effect of ash, on the other hand, is more than that of a "diluent," for additional lime and carbon are required to flux it. The effect of variation in the physical qualities of coke is more difficult to assess, but it has been established that, for Midland cokes, the 1½-in. shatter index (due regard being paid to the ½-in. index and the Cochrane abrasion index) provides a satisfactory measure of variation in hardness of any particular make of coke. Since variation in other physical properties (*e.g.* size) may affect the value of the coke in a blast-furnace, it is suggested that the hardness of "standard" coke should fall within a relatively wide range of 1½-in. shatter index (*e.g.* 75 to 80).

To allow for the difficulties involved in sampling, suitable periods of ascertainment would be agreed, for example a week, a month or three months, and the average analysis and quality of coke during this period would form the basis for the payment of bonuses or the exaction of penalties. In this period of ascertainment, it would be necessary for a certain number of samples to be taken so that the error involved in stating the average quality would not exceed the range of values for each class of coke, for the properties which are specified. Disputes about the correct quality

of coke bought or sold would therefore be minimised. Any errors possible through differences in methods of analysis and testing used could be minimised by supervision by an independent testing officer.

The qualities which are recommended for specification are purity and hardness, the purity being judged by the freedom from moisture and ash and the hardness by the $1\frac{1}{2}$-in. shatter index. Since ash is a more detrimental impurity than moisture, a higher percentage bonus or penalty should be adopted for deviation from the ash-content of the " standard " coke.

The aim of the specification is to enable purer and harder cokes to be supplied for blast-furnace work. Methods for controlling the moisture and ash-contents of coke leaving a coke works are well known, whilst simple methods for improving the hardness of cokes made from all except the poorest coking coals have been developed. Further methods are being investigated, particularly for poor coking coals. Since most of the methods involve capital expenditure or more expert control, bonuses for improved quality are offered to enable the extra cost to be faced.

Basis of Assessment.—*Sampling.* The selection of different classes of cokes, for each property specified, is based on the theory of errors and on tests which have established the validity of the theory for the ranges to be expected in the qualities specified. The theory has been fully discussed by Grumell and Dunningham (*British Standard No.* 403 *of* 1930), and application to problems of coke sampling has been made by Mott and Wheeler (The Commercial Sampling and Analysis of Coke, *Trans. Inst. Min. Eng.*, 1933, **86**, 111).

This work has shown that, in sampling coke, a large number of increments (or small samples) should be taken to represent the average quality of coke, the increments being spread as uniformly as possible over the consignment of coke the average quality of which it is intended to assess. An " increment " should consist, at the coke ovens, of an unbroken piece of coke of weight normally about $2\frac{1}{2}$ lb. or more ; but, at the blast furnaces, where breakage of coke may have occurred, an increment would be $2\frac{1}{2}$ lb., or more, and might consist of several pieces.

To obtain an accuracy of sampling such that, in 99 out of 100 periods of ascertainment, the moisture-content of the coke can be determined to within 1·4 per cent. of the true value, 48 increments are required in each period of ascertainment when the average moisture-content of the coke does not exceed 5 per cent. For this standard degree of error, the average moisture-content determined should be within 0·37 per cent. of the true value in 50 out of 100 periods of ascertainment.

A larger number of increments is necessary to obtain values within a standard degree of error for moisture than for ash. The

range of values chosen for each class of coke for moisture-content is therefore 1·5 per cent. whereas for ash it is 1·0 per cent.

Moisture-Content. The moisture-content of the "standard" coke could be, for example, over 4·0, under 5·5 per cent. The different classes for moisture-content would then be:

Class Im	Under 1·0 per cent.	
Class IIm	Over 1·0, under 2·5 per cent.	Bonus classes
Class IIIm	Over 2·5, under 4·0 per cent.	
Class IVm	Over 4·0, under 5·5 per cent.	Standard coke
Class Vm	Over 5·5, under 7·0 per cent.	
Class VIm	Over 7·0, under 8·5 per cent.	Penalty classes
Class VIIm	Over 8·5, under 10·0 per cent.	

Bonuses and Penalties for Moisture. Bonuses of 1½ per cent. of the delivered price of "standard" coke would be payable for each class above Class IVm, the coke of standard moisture-content. Similarly, penalties of 1½ per cent. of the delivered price of standard coke would be exacted for each class below Class IVm, as follows:

	Average Moisture-Content per cent	Bonus or Penalty	
Im	Under 1·0	+4·5 per cent.	of delivered price of standard coke
IIm	Over 1·0, Under 2·5	+3·0 per cent.	
IIIm	Over 2·5, Under 4·0	+1·5 per cent.	
IVm	Over 4·0, Under 5·5	Standard coke	Basic price
Vm	Over 5·5, Under 7·0	−1·5 per cent.	of delivered price of standard coke
VIm	Over 7·0, Under 8·5	−3·0 per cent.	
VIIm	Over 8·5, Under 10·0	−4·5 per cent.	

Ash-Content. The classes of coke for ash-content could be as follows:

Class Ia	Over 5·0, under 6·0 per cent.	
Class IIa	Over 6·0, under 7·0 per cent.	Bonus classes
Class IIIa	Over 7·0, under 8·0 per cent.	
Class IVa	Over 8·0, under 9·0 per cent.	
Class Va	Over 9·0, under 10 per cent	Standard coke
Class VIa	Over 10·0, under 11·0 per cent.	
Class VIIa	Over 11·0, under 12·0 per cent.	Penalty classes
Class VIIIa	Over 12·0, under 13·0 per cent.	
Class IXa	Over 13·0, under 14·0 per cent.	

Bonuses and Penalties for Ash. Bonuses of 2 per cent. of the delivered price of standard coke would be payable for each class above Va, the coke of standard ash-content. Similarly, penalties of 2 per cent. of the delivered price of standard coke would be exacted for each class below Va, as follows:

Class	Average Ash-Content (per cent.)	Bonus or Penalty	
Ia	Over 5·0, under 6·0	+8·0 per cent.	of delivered price of standard coke
IIa	Over 6·0, under 7·0	+6·0 per cent.	
IIIa	Over 7·0, under 8·0	+4·0 per cent.	
IVa	Over 8·0, under 9·0	+2·0 per cent.	
Va	Over 9·0, under 10·0	Standard coke	Basic price
VIa	Over 10·0, under 11·0	−2·0 per cent.	of delivered price of standard coke
VIIa	Over 11·0, under 12·0	−4·0 per cent.	
VIIIa	Over 12·0, under 13·0	−6·0 per cent.	
IXa	Over 13·0, under 14·0	−8·0 per cent.	

Bonuses and Penalties for Hardness. It is suggested that only small bonuses or penalties should be payable over a range of 1½-in. shatter-index of over 70 to under 82, so that the price paid for present qualities of coke would not be influenced appreciably, but inducement would be given to improve the quality. The ranges of 1½-in. shatter-index and the bonuses and penalties could be as follows:

Class Is	Over 84	+8 per cent.	of oven price of standard coke
Class IIs	Over 82, under 84	+5 per cent.	
Class IIIs	Over 80, under 82	+2 per cent.	
Class IVs	Over 75, under 80	Standard coke	Basic price
Class Vs	Over 70, under 75	− 2 per cent.	of oven price of standard coke
Class VIs	Over 65, under 70	− 10 per cent.	

Due regard should be paid to the ½-in. shatter index, which should not be less than 96·0.

Bonuses and penalties for hardness should be payable on the oven price of coke, since the hardness does not influence transport costs.

Final Assessment of Coke Quality. The average moisture and ash-contents and shatter-index of a coke should be the average of the results obtained, for a sufficient number of increments, at the coke-ovens and blast-furnaces. Any notable differences in the values obtained for the same coke at the two places of testing should be investigated by an independent testing officer to ensure that the methods of sampling and analysis are satisfactory.

Details for Sampling for Moisture and Ash Content. The essential requirement is that not less than 48 increments, each of not less than 2½ lb., should be taken in each period of ascertainment. The analysis of this number of increments would give the average moisture-content to the degree of accuracy required. These increments would not all be taken in one sampling operation, but would be spread over a period of ascertainment, namely, one week, one

month, or three months, according to convenience. At the coke-oven plants and at blast-furnace plants dealing with a small number of different cokes, a weekly period of ascertainment is convenient, but at blast-furnace plants receiving a large number of different brands in large consignments, a period of one month or more is desirable.

The moisture-content of the coke which is to be ascertained is not necessarily the true or average moisture-content of the consignment, but is the moisture-content, uninfluenced by rain or snow, for which the coke-maker is responsible. It is, therefore, necessary for coke received at the blast-furnace to be sampled only in the lower half of a wagon.

The increments should be spread as uniformly as possible over consignments of coke, and, to ensure this, the number of increments which may be taken from a wagon, or from the equivalent amount of coke on a conveyor at the coke-ovens, is limited.

The moisture-content of the daily sample must be determined as soon as possible after sampling and the results recorded for averaging when the necessary number of increments has been taken. The testing of a few daily increments will not usually give an accurate figure for the daily moisture-content, and significance should only be attached to the average value for a large number of increments.

Schemes suitable for sampling at the coke-ovens and blast-furnaces are as follows:

At the Coke Ovens.—(*i*) *Period of Ascertainment.* The period of ascertainment should be one week.

(*ii*) *The Weight of Each Increment.* The weight of each increment should be not less than 2½ lb., and would usually consist of a full-length piece of coke. Coke should not be broken in the taking of the increment and if a consignment usually contains many pieces of 5 lb. or more in weight, a proportion of these pieces should be included.

(*iii*) *The Number of Increments.* Not less than 48 increments should be taken in one week, 7 increments preferably being taken each day. Not more than 5 increments should be taken from one wagon, or the equivalent amount of coke on a conveyor, and the increments should, preferably, be spread over a number of wagons or their equivalent.

(*iv*) *The Point of Sampling.* The increments should be collected from the tops of wagons at distances corresponding to about one-sixth of the diagonal lines drawn from the corners, or their point of intersection. If less than 4 increments per wagon are taken, the points of sampling should be varied regularly from wagon to wagon. The aim of this method of taking increments is to avoid bias, in selecting particular pieces of coke. Similar rules can be applied to sampling from conveyors, a piece being taken at regular intervals according to the speed and capacity of the belt.

At the Blast-furnaces.—(*i*) *Period of Ascertainment and Increments.* The period of ascertainment should be one week, one month, or three months according to the numbers of brands of coke received and their amounts. The weight of each increment should be 2½ lb. or more, but might consist of large and small pieces produced by breakage. The number of increments should be 48 for the period of ascertainment arranged. Since a number of cokes would usually have to be sampled, the number of increments taken per wagon might be increased to 10.

(*ii*) *The Point of Sampling.* The increments should be taken from the lower halves of the wagons as they are discharged. Suitable sampling points, 2 ft. from the sides and 1 ft. from the floor, could be selected when bottom or side doors have been dropped and the coke from the centre of the wagon has been removed, leaving a wall of coke at each end.

Details of Analysis for Moisture and Ash Content. The increments taken daily must be analysed for moisture-content immediately. The methods for preparing the increments for analysis and the methods of analysis are given in the British Standard Specification for the Sampling and Analysis of Coke, the only special features being as follows:

The daily increments should be crushed so as just to pass a ½-in. square mesh screen, and a sample of not less than 4 lb. should be taken by not less than 20 increments for moisture determination. This sample (approximately 2000 grammes) should be halved and dried, the loss of moisture from one, or the average of both portions, being recorded to one decimal place, Berenger scales being used. The weighted average for at least 48 increments of 2½ lb. is the average moisture content of the coke.

The dried coke should be stored until the other daily increments have been similarly examined, when the whole of the dried coke should be mixed and a 4-lb. sample taken by not less than 20 increments for the determination of ash. This sample should be crushed on a manganese steel plate to pass a ⅛-in. mesh, using a stamp with a 5-ft. handle. A sample of 4 oz., taken by not less than 10 increments from the material crushed to ⅛-in., should be finally crushed in a manganese steel mortar with a long-handled pestle to pass a 72 B.S. mesh sieve for ash-determination.

The ash-content determined in this way would be the average ash-content during the period of ascertainment, but the ash-content of a number of daily increments might be determined separately, if desired, and the results, reported to one decimal place, averaged. The analysis of 20 increments would give the ash-content correct to ±1·0 per cent. in 99 out of 100 periods.

Details of Sampling for Shatter Testing. To obtain the best conditions for agreement between shatter test figures at the coke works and the blast-furnaces, the samples of coke should be taken from the tops of wagons at the coke-ovens or blast-furnaces or

from conveyors at the coke-ovens. To give an accuracy of ±1·0 per cent. in 99 out of 100 periods of ascertainment, about 15 shatter tests would have to be averaged for moderately hard Midland cokes, 20 tests for weaker ones, and 25 tests for the weakest cokes, in each period of ascertainment.

Not more than 6 pieces (increments of 2½ lb. or more) should be taken from each wagon or per 10 tons of coke on a conveyor. The various increments should be spread as uniformly as possible over the consignments.

The shatter tests should be made in accordance with British Standard Specification for the Sampling and Analysis of Coke. The 1½-in. shatter index for each test should be reported to 1 decimal place and the results of the requisite number of tests averaged for the period of ascertainment agreed upon. The ½-in. shatter index should also be determined and should be not less than 96·0.

Where difficulty is experienced in obtaining a sufficient number of shatter tests in any period of ascertainment, arrangement may be made to use the results obtained for the same brand of coke at other plants.

PART III
THE FORMATION OF COKE

CHAPTER XIII

THE COMPOSITION OF COAL

THE most important elements of which coal is composed are carbon, hydrogen and oxygen. There are present also small quantities of nitrogen and sulphur. Other elements, such as those contained in the "ash," are conveniently regarded as adventitious impurities.

THE "COAL BAND."

If for a number of coals the carbon and hydrogen-contents are plotted on rectangular co-ordinates, as in Seyler's classification of coals (*Proc. S. Wales Inst. Eng.*, 1900, **21**, 483; 1931, **47**, 347), the points, whether they relate to lignites at the one end of the scale or to anthracites at the other, are found to lie within a narrow band—the so-called "coal band."

The carbon-content varies over a wider range than the hydrogen-content and serves as a measure of the "rank" or "degree of coalification" of the coal. The coals of higher rank, *i.e.* of higher carbon-content, are usually older geologically than those of lower rank; and at a given colliery the seams usually lie beneath the surface in order of their rank, the deepest having the highest carbon-content. It is usually assumed that the rank of a coal is determined mainly by the pressure and temperature to which, during the period of their transformation, the coal-forming materials have been subjected by overlying strata, but sometimes abnormal earth-movements appear to have accelerated change and there may be, as in the South Wales coal-field, marked lateral alteration in the carbon-contents of the seams.

To avoid misunderstanding, the distinction between the carbon-content here referred to and the so-called "fixed carbon" of a coal should be noted. The former is elementary carbon, as determined by combustion analysis, and the latter is the residue of coke obtained when a determination of the "volatile matter" in a coal is made, *e.g.* by crucible test. The "fixed carbon" contains the elements hydrogen, oxygen, nitrogen and sulphur as well as carbon.

In Fig. 60, the carbon-contents of about 1,200 (American) coals have been plotted against their oxygen-contents, the analyses having been calculated on a dry, ash- and sulphur-free basis (H. G. Hickling, *Trans. Inst. Min. Eng.*, 1928, **72**, 261). Within a narrow

Land, there is a nearly straight line relationship for all the coals up to about 90 per cent. carbon, so that the oxygen-content, which is sometimes taken as an index of the coke-making value of a coal (*e.g.* H. J. Rose, *Amer. Inst. Min. Eng.*, 1926, see *Fuel in Science and Practice*, 1926, **5**, 569), could be replaced by the carbon-content, which, unlike the oxygen-content, can be directly determined by analysis.

Fig. 61 illustrates in a similar way the relationship between carbon and hydrogen (Parr's basis) derived by L. W. Needham

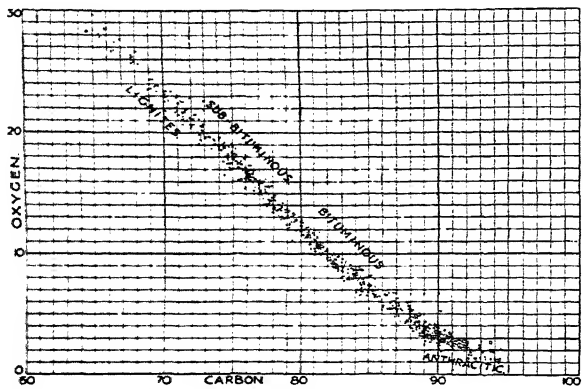


FIG. 60.—The Relationship between Carbon and Oxygen Contents of Coals, Lignites to Anthracites (Hickling).

(private communication) for 1,000 representative coals from different parts of the world. Iso-vols (lines indicating the same volatile matter content) and iso-cals (lines indicating the same calorific value) have been drawn on this graph. It will be seen that the iso-vols cut across the coal band rather acutely above about 87 per cent. carbon. Whilst the volatile matter, therefore, can serve as a distinguishing characteristic for coals containing less than about 30 per cent., it ceases to be, by itself, of much value at about 35 per cent., for along this iso-vol the carbon-content of the coal may vary between 83 and 88 per cent., and the properties of coals within this range of carbon-content differ rather widely. For the same reason, the iso-cals, by themselves, do not serve to distinguish between coals of high carbon-content.

The Classification of Coals

The volatile matter is used as a means of grouping coals by the Belgian Railways Administration, the groups being: "Lean" coals, 8½ to 11½; "quarter-fat," 11½ to 13; "half-fat," 13 to 18; and "fat," over 18 per cent. volatile matter. Similarly, Gruner's (1874) well-known classification (Table LXVIII) is based essentially

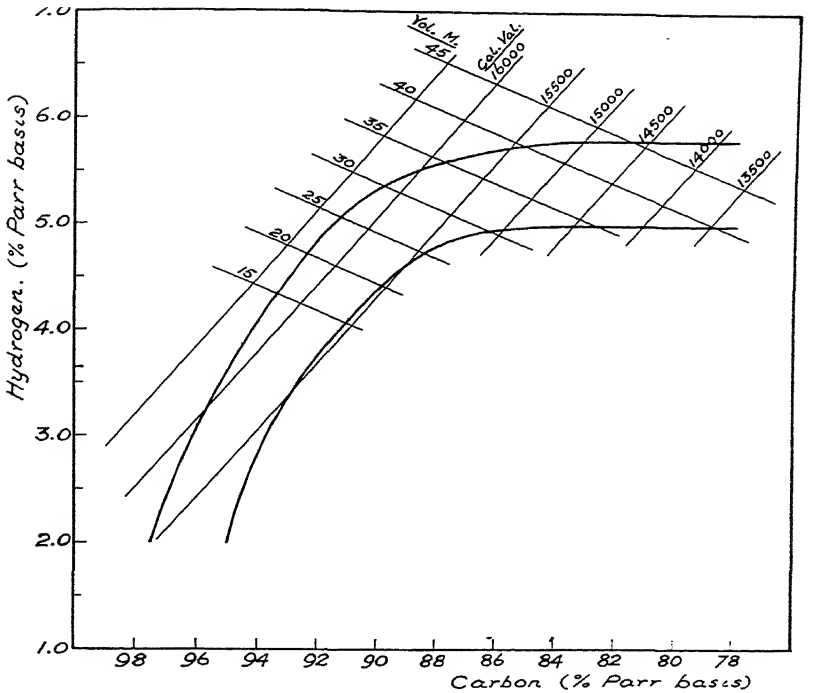


FIG. 61.—The Relationship between Carbon and Hydrogen Contents of Coals, Lignites to Anthracites (Needham).

on the volatile matter contents of the coals. (*Atlas général des houillères, II,* 1911.)

TABLE LXVIII.—GRUNER'S CLASSIFICATION OF COALS.

Group	Volatile Matter, Per cent.	Carbon, Per cent.	Hydrogen, Per cent.
Lean, or anthracitic . .	10 to 18	93 to 90	4·0 to 4·5
Short-flame fat, or coking .	18 to 26	91 to 88	4·5 to 5·5
True fat, or smith's . .	26 to 32	89 to 84	5·0 to 5·5
Long-flame fat, or gas-making	32 to 40	85 to 80	5·0 to 5·8
Dry long-flame . . .	40 to 45	80 to 75	4·5 to 5·5

The range of composition, 18 to 26 per cent. volatile matter, suggested for coals for coke-making in Gruner's classification, if applied to British coals, would rule out most of those at present used except the South Wales coking coals, which resemble the

Continental coals most closely; the range is undoubtedly the most suitable for the production of hard metallurgical coke, but the yields of gas, tar and benzole are less satisfactory than from coals of lower rank.

In America, tentative standards for the classification of coals according to their rank were approved by the American Society for Testing Materials in 1935, as in Table LXIX.

TABLE LXIX.—A.S.T.M. CLASSIFICATION OF COALS ACCORDING TO RANK.

Class	Group	Limits of Volatile Matter	Calorific Value, B.Th.U. per lb.	Physical Properties
Anthracite	Meta-anthracite	< 2		Non-agglomerating.
	Anthracite	2– 8		
	Semi-anthracite	8–14		
Bituminous	Low volatile bituminous	14–22		
	Medium volatile bituminous	22–31		
	High volatile—			
	bituminous A	> 31	> 14,000	Either agglomerating or non-weathering.
	bituminous B		14,000–13,000	
	bituminous C		13,000–11,000	
Sub-bituminous	Sub-bituminous A		13,000–11,000	Weathering and non-agglomerating
	,, B		11,000– 9,500	
	,, C		9,500– 8,300	
Lignite	Lignite		< 8,300	Consolidated
	Brown coal			Unconsolidated.

NOTE.—Both volatile matter and calorific value are calculated on a mineral-matter-free basis, the volatile matter on the dry coal and the calorific value on the moist coal, *i.e.* containing natural pit moisture, but not including visible moisture on the surface.

The calculation of calorific value on the "moist" coal in this American classification is intended to take account of the wide differences that there are in the natural moisture-contents of American high-volatile bituminous coals, but this part of the classification is unsatisfactory. For, when ultimate analyses of typical "high-volatile bituminous coals A, B and C" are examined, it is seen that class A may include coals varying in carbon-content from 82 to 88 per cent. (on a dry and mineral-matter-free basis), class B may contain 81 to 84 per cent. of carbon, and class C 80 to 83 per cent. Each of these ranges of carbon-content, which overlap in the three classes, includes coals which may vary widely in their properties.

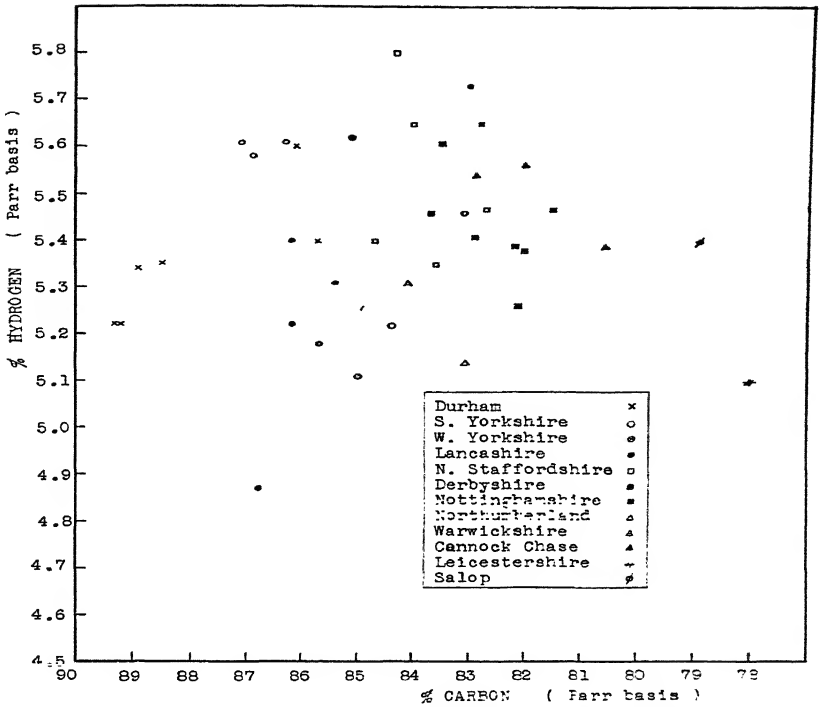
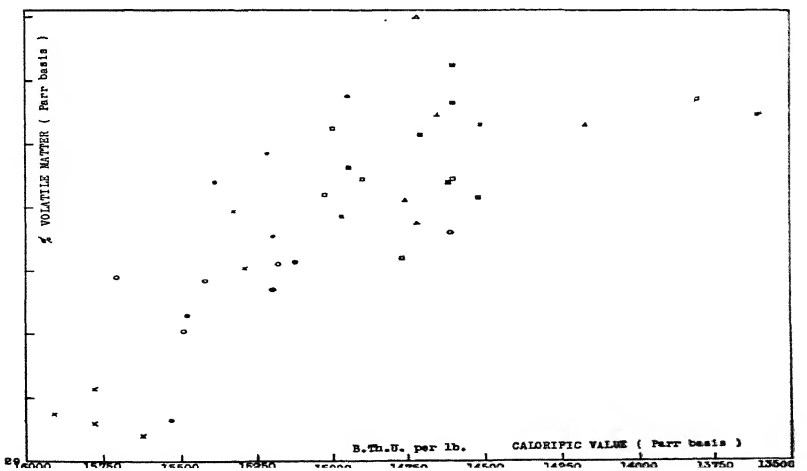


FIG. 62.—The Relationship Carbon to Hydrogen for Typical British Coals.



In Great Britain, most of the coals are high in volatile matter, which, for such coals, for the reason already given, does not distinguish satisfactorily between different types. The carbon-content, however, as used by Seyler, provides a good basis for classification. Unfortunately, there has not yet been accumulated a sufficient

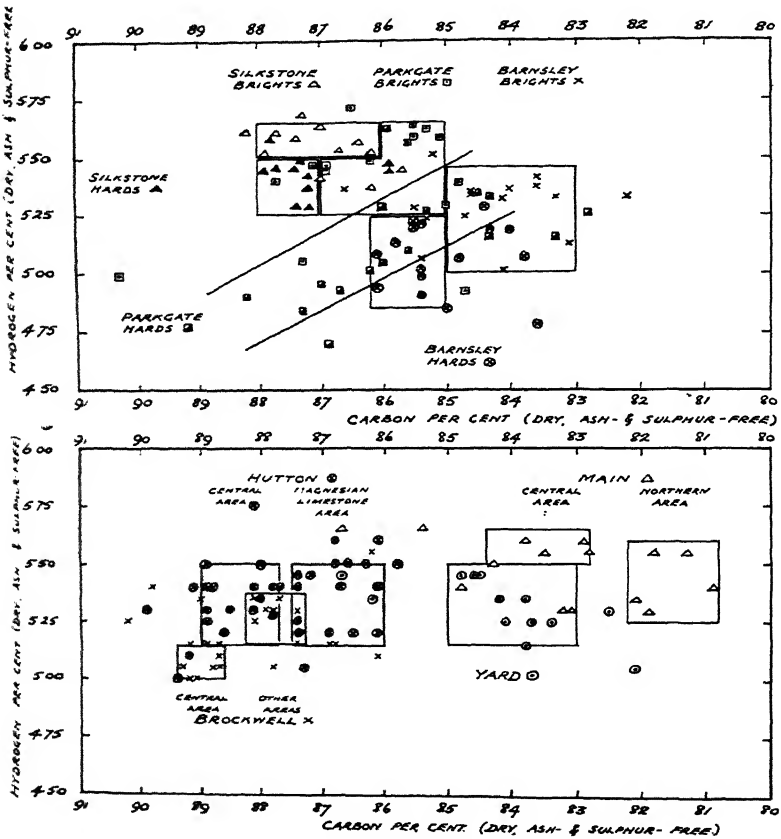


FIG. 64.—The Variation of Carbon and Hydrogen in Different Sections of Seams in Yorkshire (above) and Durham and Northumberland (below).

number of reliable ultimate analyses of coals, outside the South Wales coalfield, although the Fuel Research Board's Survey is gradually providing them. In Table LXX, analyses of typical British coals have been collated from the Survey reports, together with a number supplied by E. S. Grumell (*Trans. Inst. Min. Eng.*, 1931, **81**, 214) and some of our own. For each coalfield the

I. DURHAM
- Low Main . . .
- Maudlin . . .
- Hutton . . .
- Ballarat . . .
- Busty . . .
- Brockwell . .

II. SOUTH YORKSHIRE
- High Hazel . .
- Barnsley (bright) .
- Parkgate . .
- Silkstone . .
- Halifax Soft . .

III. WEST YORKSHIRE
- Haigh Moor . .
- Upper Beeston . .

IV. DERYBSHIRE
- Top Hard . .
- Deep Soft . .
- Deep Hard . .
- Tupton . . .
- Black Shale . .

V. NORTH STAFFS.
- Main . . .
- Ash . . .
- Holly Lane . .
- Banbury 7 ft. . .
- Cockshead . .

VI. LANCASHIRE
- Pemberton 5 ft. .
- Wigan 4 ft. . .
- Trencherbone .
- Ravine . .
- Arley . .
- Lower Mountain Min

VII. NORTHUMBERLAN
- Main . .
- Yard . .
- Low Main .

VIII. NOTTINGHAMSHI
- High Hazel .
- Top Hard .
- Deep Soft .
- Low Main .

IX. WARWICKSHIRE
- Thick . .

X. CANNOCK CHASE
- Eight Feet .

XI. LEICESTERSHIRE
- Main . .

XII. SHROPSHIRE
- Clod . .

seams are given in descending order in which they lie beneath the surface.

The districts to which these analyses of typical coals relate are responsible for about 75 per cent. of the coal-production of Great Britain. Only about 17 per cent. of the output is from South Wales, where the volatile matter contents might be used satisfactorily as a basis for classification, and the majority of British coals fall on the horizontal portion of the "coal band" shown in Fig. 61. In Fig. 62, carbon-contents are plotted against hydrogen-contents for the coals of which the analyses are given in Table LXX. It will be seen that the variation in hydrogen-content is small, whilst the greatest concentration lies within the range 82–84 per cent. carbon. In Fig. 63, volatile matter is plotted against calorific value (on rectangular co-ordinates) for the same coals.

In Fig. 64 the "coal band" is divided up into zones indicating the limits of carbon and hydrogen for a number of seams exemplified by numerous pillar sections, of which analyses are available through the Fuel Research Board's Survey. For the analyses of the Yorkshire coals, the analyses of the "hards" (durain), which occur in wide bands which differ in composition from the "brights" (clarain and vitrain) with which they are associated, have been excluded. In any particular area, it is evident that a seam does not vary much in composition, so that each seam can be characterised by its average composition.

The Relationships between Ultimate Analysis and Volatile Matter and Calorific Value.—The relationships between the carbon, hydrogen and volatile matter contents of coals and their calorific values are important. They have not received the attention they merit, no doubt owing to the shortage of data for representive coals. C. A. Seyler (*J. Soc. Chem. Ind.*, 1933, **52**, 304T) has deduced a relationship between volatile matter and carbon and hydrogen contents, all expressed on Parr's "unit coal" basis (*Bull.* 180, *Univ. of Ill. Exp. Stat.*, see *Fuel in Science and Practice*, 1937, **16**, 52), which is a refinement on calculations on the moisture, ash and sulphur-free basis, in which allowance is made for the difference between "mineral matter" and "ash." The relationship, V, H and C being the percentages of volatile matter, hydrogen and carbon, is:

$$V = 10{\cdot}593H - 1{\cdot}227C + 83{\cdot}54 \quad . \quad . \quad . \quad . \quad . \quad (1)$$

According to Dulong's formula:

$$Q_g = 145{\cdot}4C + 620{\cdot}3H - 45{\cdot}0S$$

where Q_g is the gross calorific value in B.Th.U. per lb. and C, H and S are the percentages of carbon, hydrogen and sulphur (on a dry, ash-free basis). On the assumption that the average nitrogen-content of coal is 1·0 per cent. (a rather low estimate), Seyler has

deduced a relationship between the calorific value (B.Th.U. per lb.) and analyses on "unit coal" basis, as follows:

$$Q_g = 224 \cdot 1(C + 3 \cdot 12\ H - 34 \cdot 3) \qquad (2)$$

Table LXXI illustrates the variations in calorific value of coals according to their carbon and hydrogen contents (on "unit coal" basis), calculated from Seyler's equation (2).

TABLE LXXI.—THE RELATIONSHIP BETWEEN THE CALORIFIC VALUE AND THE CARBON AND HYDROGEN CONTENTS OF COALS. (PARR'S "UNIT COAL" BASIS).

Carbon per cent.	Calorific Value, B.Th.U. per lb.			
	4·75% H	5·0% H	5·25% H	5·5% H
80·0	13,550	13,750	13,900	14,100
82·5	14,100	14,300	14,480	14,630
85·0	14,620	14,950	15,100	15,200
87·5	15,220	15,420	15,580	15,750
90·0	15,790	15,980	16,150	16,300

There are some exceptions to Seyler's equation 1, but it is useful in drawing attention to the dependence of the volatile matter content of a coal on its hydrogen-content. The effect of variation in the hydrogen and carbon contents of coals on their volatile matter contents, calculated from Seyler's equation 1, is illustrated in Table LXXII.

TABLE LXXII.—THE RELATIONSHIP BETWEEN THE VOLATILE MATTER AND THE HYDROGEN AND CARBON CONTENTS OF COALS. (PARR'S "UNIT COAL" BASIS).

Carbon, per cent.	Volatile Matter, per cent.			
	4·75% H	5·0% H	5·25% H	5·5% H
80·0	35·7	38·3	41·0	43·6
82·5	32·7	35·3	38·0	40·6
85·0	29·7	32·3	35·0	37·6
87·5	26·7	29·3	32·0	34·6
90·0	23·7	26·3	29·0	31·6

COKING COALS

Only the coal with carbon 90 per cent. and hydrogen 4·75 per cent. in Table LXXII conforms with Gruner's classification for a "coking coal." On the Continent and in South Wales it is common practice to blend coals of high volatile matter content with a coal

low in volatile matter (containing less than 20 per cent.), so as to produce a blend containing about 23 per cent. Later in this section (Part III), it will be shown that the formation of coke occurs in two principal stages which may be termed "agglomeration" and "devolatilization." The volatile matter of a coal does not, *per se*, influence the stage of agglomeration, but during the stage of devolatilization the more volatile matter there is then present the greater is the degree of fissuring of the rigid semi-coke.

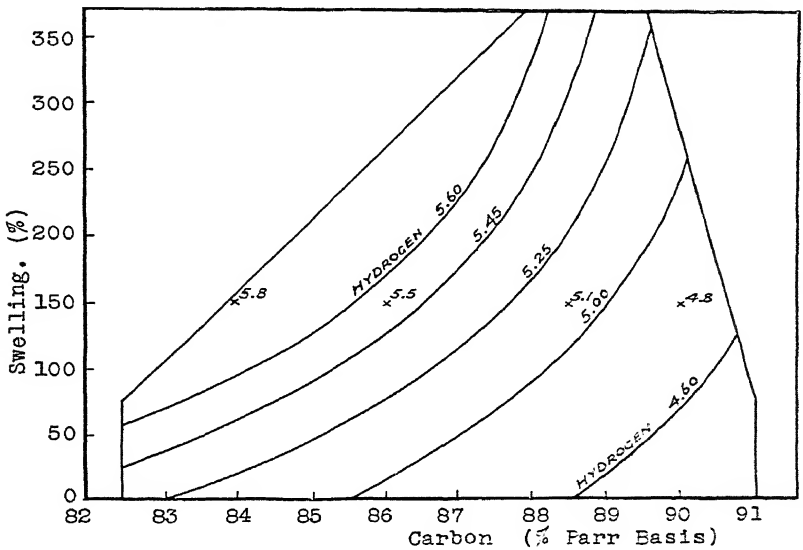


FIG. 65.—The Relationship between Swelling-power and Carbon and Hydrogen Contents.

SWELLING-POWER

The first stage of coke-formation, agglomeration of the charge, is dependent on a property of coal which can conveniently be measured, as in the Sheffield Laboratory Coking Test, by its swelling-power during heating. Fig. 65 illustrates the relationship between the swelling-powers of coals and their carbon and hydrogen contents. No coal containing less than 82·5 or more than 91* per cent. of carbon swells during heating, so that only coals of carbon-contents within that range can be used, by themselves, for coke-making. The magnitude of the swelling-power is dependent not only on the carbon-content but on the hydrogen-content, increasing with increase in carbon-content, particularly so above 86 per cent.

* Unless otherwise stated, all analyses are expressed on Parr's dry and mineral-matter-free basis, an explanation of which is given on p. 352.

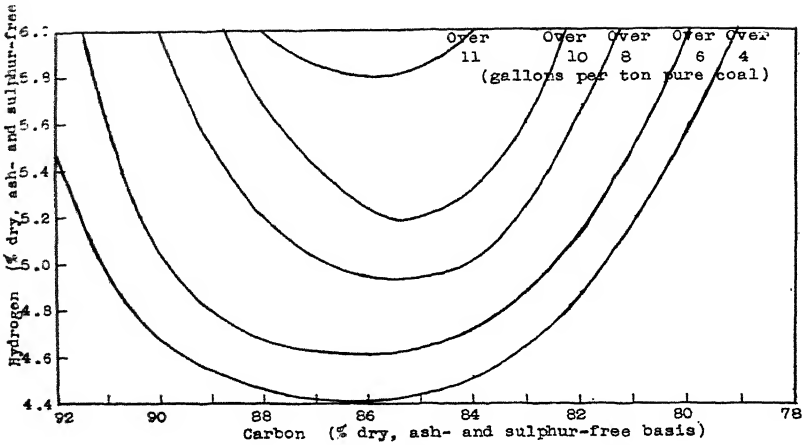


FIG. 66.—Lines of Equal Tar Yields for Canadian Coals of Different Carbon and Hydrogen Contents. Experimental Coke Oven.

carbon, and, for a given carbon-content, increasing also with increase in hydrogen-content. On Fig. 65 four coals have been marked, each having a swelling-power of 150 per cent., of carbon-contents 90, 88·5, 86 and 84 per cent. Their hydrogen-contents are 4·8, 5·1, 5·5 and 5·8 per cent., and they represent a South Wales, a Durham and a Yorkshire coking coal, and one which is not typical

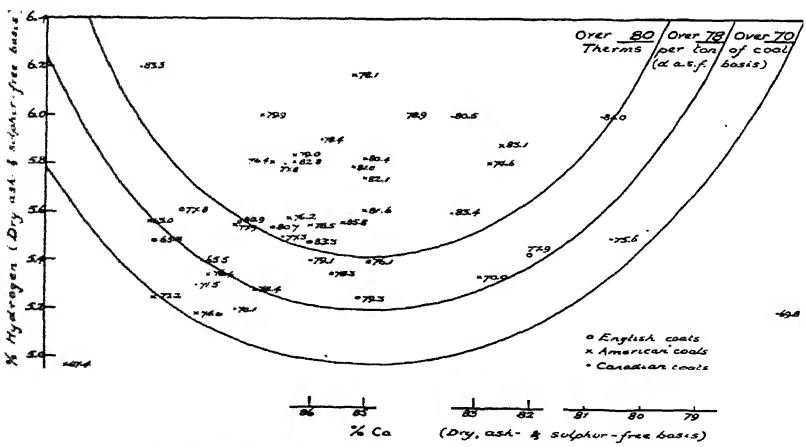


FIG. 67.—The Relationship between Thermal Yields of Gas and Carbon and Hydrogen Contents of Coal.

of any particular district. The volatile matter contents, as calculated by Seyler's equation 1, would be 23·8, 29·0, 36·5 and 41·9 per cent. Each of these coals would behave satisfactorily for coke-making during the stage of agglomeration, but during the stage of devolatilization fissuring would become greater, and the coke correspondingly weaker, the higher the volatile matter content.

High swelling-power is a valuable quality for a coking coal, the more so, for the production of coke of high impact hardness, if it is associated with a low volatile matter content. On the other hand, a coal of high volatile matter content is rich in hydrogen, and in by-product coking the yields of gas, tar and benzole from it will be high. A high thermal yield of gas is of value not only at coke-oven plants at collieries, when the gas is sold, but at plants at iron and steel works, when use is made of the gas as a general fuel; whilst the yields of tar and benzole also affect the economy of the coke-making process. In part, therefore, and within reason, inferior coke-making qualities of a coal may be compensated by superior yields of by-products.

The Relationships between Ultimate Analysis and Yields of By-Products

Tar.—Data are not available for British coals to show the relationship between ultimate compositions and yields of tar, but some have been obtained by the Department of Mines of Canada for Canadian coals and are given in Fig. 66. The yields of tar were from 2-ton charges in an experimental oven and are expressed in imperial gallons per ton of dry, ash- and sulphur-free coal. It will be seen that lines of approximately equal tar-yield can be drawn on the diagram, indicating maximum yields from coals containing between 84 and 88 per cent. of carbon. The effect of the hydrogen-content is clearly shown,* the yield of tar being greatest from coals of high hydrogen-content and dropping rapidly when the hydrogen falls below 5·2 per cent.

Thermal Yields of Gas

In Fig. 67, the yields, in therms, of gas from Canadian coals, per ton (2,240 lb.) of dry, ash- and sulphur-free coal, are recorded on rectangular co-ordinates of carbon and hydrogen contents. The lines of equal gas-yield show that the maximum is obtained from coals of about 85 per cent. carbon, but the variation is small over the range 82 to 87 per cent. The hydrogen-content of the coal has a marked influence on the gas-yield, a fact which is not surprising when it is remembered that hydrogen and methane form

* Recent work by the Midland Coke Research Committee, has shown that the tar-yields obtained from coals under similar conditions of heating can be expressed by the formula $K(H - 4{\cdot}20)$ per cent., the coefficient K having a value of 11 to 5 (or less) according to the temperature and the degree of cracking, and H being the hydrogen content.

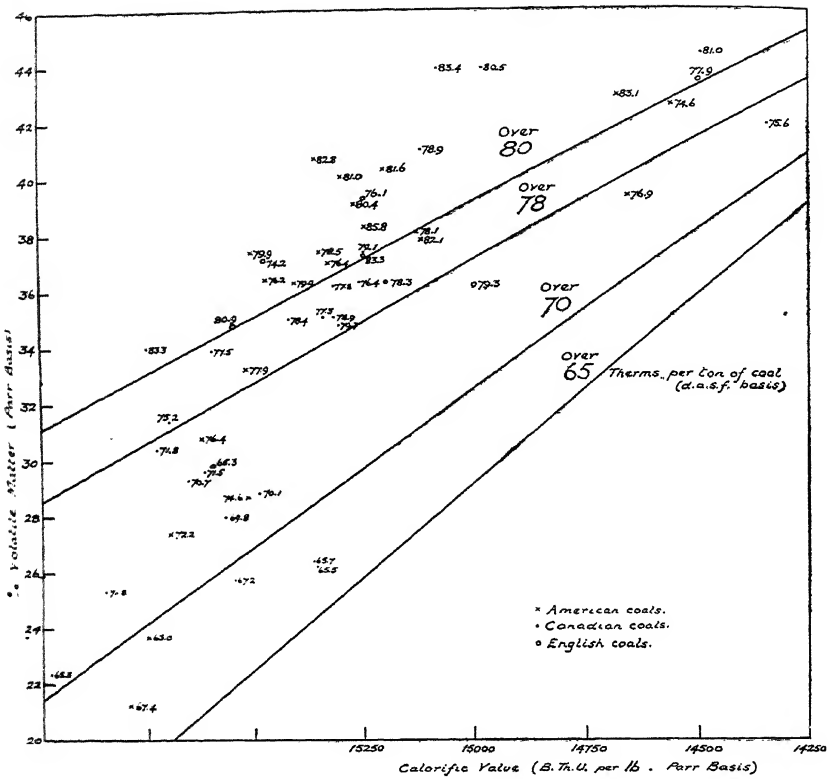


FIG. 68.—The Relationship between Thermal Yields of Gas and Volatile Matter and Calorific Value of Coal.

75 per cent. of coke-oven gas. On the same graph, a number of records for British coals have been included, based on values in reports by the Fuel Research Board in which the yields of gas as obtained by the Gray-King high-temperature assay are given. The yields are in a few instances higher than for Canadian coals of the same composition, but are otherwise similar. The Gray-King high-temperature assay has also been used by the United States Bureau of Mines for a series of 22 coals ("Gas, Coke and By-Product Making Properties of American Coals and their Determination" by A. C. Fieldner and J. D. Davis, *Monograph No.* 5, 1934), and the gas-yields obtained fit satisfactorily into Fig. 67 with but few exceptions. Light oil yields obtained by the last-named writers

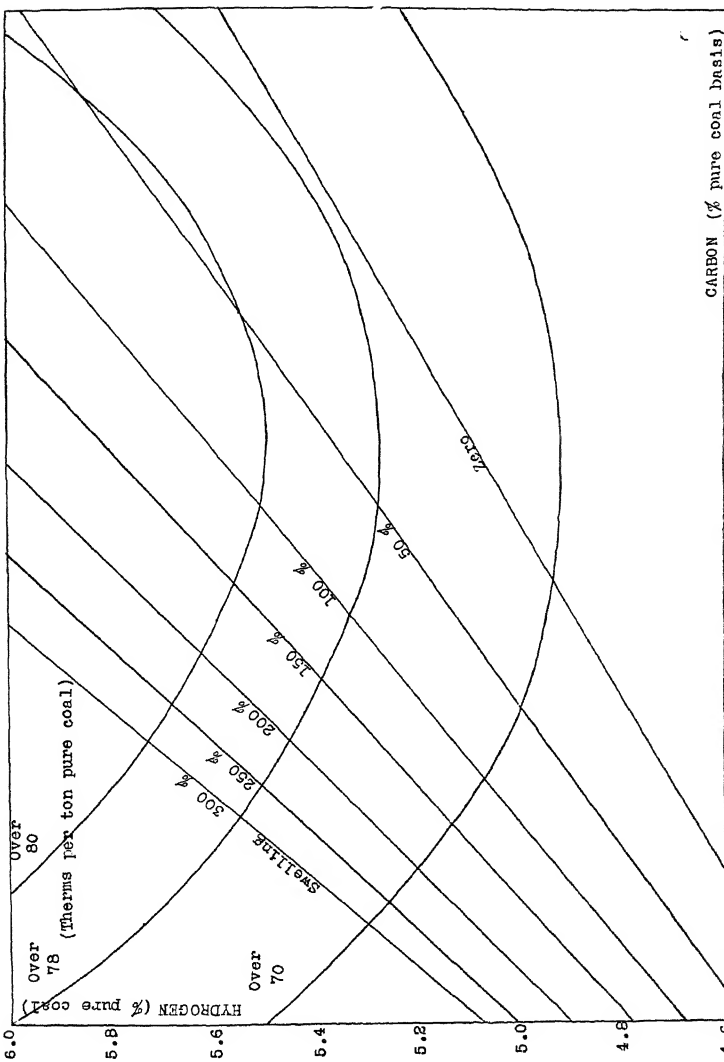


FIG. 69.—The Relationship between Thermal Yields of Gas, Swelling Power and Carbon and Hydrogen Contents of Coal.

in the B.M.—A.G.A. assay test at 900° C. can be correlated in a graph similar to Fig. 66.

In Fig. 68, the yields of gas are plotted with calorific values and volatile matter contents as co-ordinates.

These results in general show the importance of the hydrogen-content of a coal with respect to its yields of by-products during coking. Since 1 per cent. of hydrogen has a greater influence on the calorific value of a coal than 1 per cent. of carbon, the calorific value becomes a good index of the probable yield of by-products, particularly if the volatile matter content is also known. Reference to Fig. 68 shows that coals of which the calorific value exceeds 14,600 B.Th.U. per lb. and the volatile matter 34 per cent. give the highest yields of gas.

TABLE LXXIII.—THE RELATIONSHIP BETWEEN THE ANALYSES OF COALS AND THE QUALITY OF THEIR COKES.

Coal	Analysis of Coal				Quality of Coke				
	Carbon	Hydrogen	Vol. Matter	Swelling Power, per cent.	Shatter Indices 1½-in.	Shatter Indices ½-in.	Apparent Density	Real Density†	Total Pores, per cent.
Two Foot Nine (S. Wales)	89·7	4·65	21·9	145	97	99·0	0·83	1·93	52·5
Victoria (Durham)	89·7	4·82	24·6	134	95	98·6	0·88	1·96	55·9
Busty (Durham)	88·7	4·93	25·6	199	93	98·1	0·95	1·80	49·3
No. 5 (Kent)	87·5	5·20	31·0	126	88	98·4	0·95	1·87	50·8
Parkgate (S. Yorks.)	86·9	5·40	36·7	129	89	98·1	1·00	1·83	46·8
Halifax Soft (S. Yorks.)	86·8	5·35	33·0	136	92	97·9	1·01	1·91	49·5
Mountain Mine (Lancs.)	86·6	5·39	30·3	86	92	97·9	1·01	1·87	48·3
Silkstone (S. Yorks.)	86·5	5·45	35·1	100	86	97·3	0·88	1·91	56·1
Barnsley (S. Yorks.)	85·1	5·17	34·5	65	79	96·9	0·77	1·75	57·2
Winter (W. Yorks.)	83·9	5·12	33·7	8	70	97·6	0·86	1·71	50·8
Waterloo (Derbyshire)	83·0	5·35	36·2	nil	72*	95·6	0·92	1·70	48·3
Black Shale (Derbyshire)	82·8	5·33	37·8	12	67*	95·6	0·93	1·78	49·7
Tupton (Derbyshire)	82·6	5·28	36·8	13	72*	97·4	0·90	1·80	51·3
Deep Soft (Derbyshire)	82·6	5·31	35·1	19	72*	96·7	0·96	1·71	46·4
Swallow Wood (S. Yorks.)	82·3	5·40	36·8	nil	72	96·4	0·74	1·71	58·0

* Compressed charges. † Calculated on an ash-free basis.

In Fig. 69, lines of equal thermal yields of gas are superposed on lines of equal swelling-power, percentages of carbon and hydrogen in the coals being the co-ordinates. A similar graph may be drawn with volatile matter and calorific value of the coal as co-ordinates. It will be seen that coals of 84 to 85 per cent. carbon and 5·5 per cent. hydrogen (or coals of volatile matter content 35 per cent. and calorific value 15,000 B.Th.U. per lb.) are of 50 to 100 per cent. swelling-power, a value sufficient to ensure satisfactory physical properties of the cokes.

When the swelling-power of a coal is low, as it is for coals of less than 83 per cent. carbon and of moderate hydrogen-content, the agglomeration of the individual particles in a charge in a coke-oven is insufficient to produce what is sometimes termed a "well-fused" coke, and the coke is abradable. Moreover, when a British coal is of low rank (low carbon content) it is usually of high volatile matter content and the coke it yields is so fissured as to be of low impact hardness. This is illustrated by the records in Table LXXIII which are of tests of cokes made from single seams of coal in full-scale ovens.

TABLE LXXIII contains values for the densities of the cokes, from which it appears that the real density falls with the rank of the coal from which the coke is made. With coals of 82·5 to 84 per cent. carbon, which are almost non-swelling in the Sheffield Laboratory Coking Test and may produce abradable cokes, the shatter indices are low, and it was considered necessary in practice to compress the charges to produce commercial cokes, carbonisation being carried out in wide ovens with a long time of coking.

BANDED BITUMINOUS COALS

Most British seams of coking coals are banded, the major constituent being clarain, which, together with a small proportion of vitrain, constitutes the "brights." Durain ("hards") is frequently inter-banded with the clarain and sometimes, as in the Midlands and in Scotland, forms a sufficiently distinct and important constituent to be separable commercially from the brights. This is so, for example, with the Silkstone, Parkgate and Barnsley seams, the hards of the last-named being marketed as locomotive and bunker coals. Fusain is but a minor (though an important) constituent, rarely forming more than 3 per cent. of the seam. Being friable, the fusain concentrates in the smaller sizes of coal and particularly in the dust.

Durain and Clarain.—The proportions of durain (and fusain) in a number of seams are recorded in Table LXXIV. For all except the Lancashire coals the values are mean values for from 12 to 24 sections of the same seams and are taken from the Fuel Research Board's Survey Papers. For the Lancashire seams the values

are those given by F. S. Sinnatt (*Trans. Inst. Min. Eng.*, 1922, **63**, 307) for single sections.

TABLE LXXIV.—THE PROPORTIONS OF DURAIN AND FUSAIN IN COAL SEAMS.

	Durain, per cent.		Fusain, per cent.
	Range of values	Mean	
Barnsley (S. Yorks.)	21–63	39	2·0
Parkgate (S. Yorks.)	15–42	26	1·4
Silkstone (S. Yorks.)	20–44	32	1·7
Hutton (Durham)	Up to 22	10	2·0
Brockwell (Durham)	Up to 42	12	4·3
Top Busty (Durham)	Up to 34	11	2·9
Bottom Busty (Durham)	Up to 34	13	2·7
Yard (Northumberland)	6–26	15	2·8
Beaumont (Northumberland)	6–38	19	2·9
Main (Northumberland)	7–42	23	2·6
Plessey (Northumberland)	20–59	39	1·7
Upper Beeston (W. Yorks.)	8–26	16	5·0 (including dirt)
Deep Hard (Derbyshire)	20–45	37	2·3
Arley (Lancs.)	18		1·4
Wigan 5-ft. (Lancs.)	35		1·9
Arley (Lancs.)	21		1·8
Wigan 3-ft. (Lancs.)	20		1·0
Queen (Lancs.)	nil		nil

The proportion of durain is high in the South Yorkshire and low in the Durham seams. The high proportion of fusain in the Brockwell seam is noteworthy.

TABLE LXXV.—AMOUNTS OF DURAIN IN SMALL COAL FROM THE BARNSLEY SEAM.

Seam Section	Durain, per cent.			Dull Coal, per cent. of seam	Middle Hards, per cent. of seam
	In Double Nuts (2-in. to 1-in.)	In Single Nuts (1-in. to ⅜-in.)	In Slack (through ⅜-in.)		
D.	40	45	47	52	65
H.M.	—	—	40	31	24
S.K.	—	—	27	34	40
H.	—	—	20	30	44
Y.M.	—	—	18	34	35
M.	35	37·5	23	—	39
R.	32	23	22	39	39

The proportion of dull coal (mainly durain) found in different grades of marketed coal does not depend solely on the proportion

in the seam as a whole, as is seen from Table LXXV, in which the amounts of durain in the smaller sizes of coal from a number of pits working the Barnsley seam are compared with the amounts in the seam as a whole at those pits.

As a rule, the percentage of dull coal in the Barnsley seam, as recorded in the Fuel Research Board's Survey Reports, is less than that of the middle band of "hards," indicating that this band is not exclusively durain. There is some durain, on the other hand, in the so-called "top softs" and "bottom softs" of the Barnsley seam (the bright portions of the seam), as is shown by the fact that in section H.M. the proportion of dull coal is greater than the proportion of middle hards. As regards the commercial grades of coal, the durain is present in greatest proportion in D (as also is the dull coal in the seam section), but it is also present in high proportion in the slack of H.M., the seam section of which contains some durain in the top and bottom softs. Except in these two instances, the durain forms about one-quarter of the slack.

An examination of the graded sizes of coal at a South Yorkshire colliery working the Parkgate and Silkstone seams showed the amounts of durain recorded in Table LXXVI.

TABLE LXXVI.—AMOUNTS OF DURAIN IN GRADED SIZES OF (MIXED) PARKGATE AND SILKSTONE SEAMS.

Grade	Size (in.) *	Per cent. of Saleable Output	Durain, per cent.
Large	Over 6	3·4	42
Cubes	6–2	9·6	35
House seconds	Over 2	2·3	—
House doubles	2–1½	6·0	—
Gas doubles	1½–1 1/8	11·0	34
Gas singles	1 1/8–11/16	13·1	26
Beans	11/16–9/16	8·2	16
Dedusted slack	Through 9/16	42·0	25
Dust	Through 60-mesh	3·6	†
Run of mine	—	—	21

* The sizes above 1½-in. are square mesh, the remainder are round mesh.
† Contains 25 per cent. fusain.

The clarain and durain of the principal seams in South Yorkshire differ in analysis in the manner shown in Table LXXVII. The values are taken from the Fuel Research Board's Survey Reports and are mean values for from 12 to 16 seam-sections. Values are also given for analyses of the "softs" and the "hards," which are the bright and dull benches of coal into which the seam divides naturally.

It will be noted that there is a greater difference between the hydrogen-contents of the clarain and the softs of the Barnsley

seam than obtains with the other two seams, indicating that in the Barnsley softs the clarain is inter-banded with some durain.

TABLE LXXVII.—ANALYSES OF CLARAINS AND DURAINS OF YORKSHIRE COAL SEAMS.

Seam	Carbon *		Hydrogen *		Volatile Matter†		Carbon *		Hydrogen *	
	Clarain	Durain	Clarain	Durain	Clarain	Durain	Softs	Hards	Softs	Hards
Silkstone .	86·9	88·1	5·60	5·59	36·5	36·8	87·0	87·2	5·53	5·42
Parkgate .	85·6	87·1	5·38	5·10	37·4	34·0	86·3	85·6	5·44	5·11
Barnsley .	83·9	85·3	5·39	4·90	38·7	34·4	84·4	85·0	5·28	5·07

* Per cent. on dry, ash- and sulphur-free basis.
† Per cent. on dry, ash-free basis.

Although the wide bands of durain to be found in most of the seams in the Yorkshire coalfield are of lower hydrogen-content than the clarains of the same seams, this is not invariably so in other coalfields. A comparison of the analyses of durains and clarains from the same seams in different coalfields, taken from the Fuel Research Board's Survey Reports, is given in Table LXXVIII. The values are mean values for three or more seam-sections.

TABLE LXXVIII.—ANALYSES OF CLARAINS AND DURAINS OF SEAMS IN DIFFERENT COALFIELDS.

Seam		Per cent. air-dried coal			Per cent. Parr's basis		
		Moisture	Ash	Sulphur	Carbon	Hydrogen	Vol. Matter
I. DURHAM							
Brockwell	Clarain .	1·2	2·4	0·55	88·8	5·10	30·2
	Durain .	0·9	13·5	0·49	88·8	5·68	32·8
Busty	Clarain .	1·4	3·3	0·87	—	—	32·3
	Durain .	1·1	4·8	0·66	—	—	31·2
Hutton	Clarain .	2·5	4·1	2·2	—	—	39·0
	Durain .	1·8	8·1	1·9	—	—	39·1
II. NORTHUMBERLAND							
Beaumont	Clarain .	5·5	3·9	1·28	—	—	38·7
	Durain .	4·1	5·6	1·44	—	—	42·1
Yard	Clarain .	4·8	1·5	0·85	83·9	5·44	38·4
	Durain .	2·7	3·3	0·68	85·1	5·33	41·7
III. LANCASHIRE							
Ravine	Clarain .	2·6	4·8	2·86	—	—	39·1
	Durain .	2·1	24·5	2·08	—	—	33·7
Arley	Durain .	1·2	3·5	1·26	—	—	34·0
	Total seam	1·1	3·2	1·44	—	—	34·1

There are, apparently, two extremes of durains, the one of relatively low hydrogen-content, as found in wide bands in the

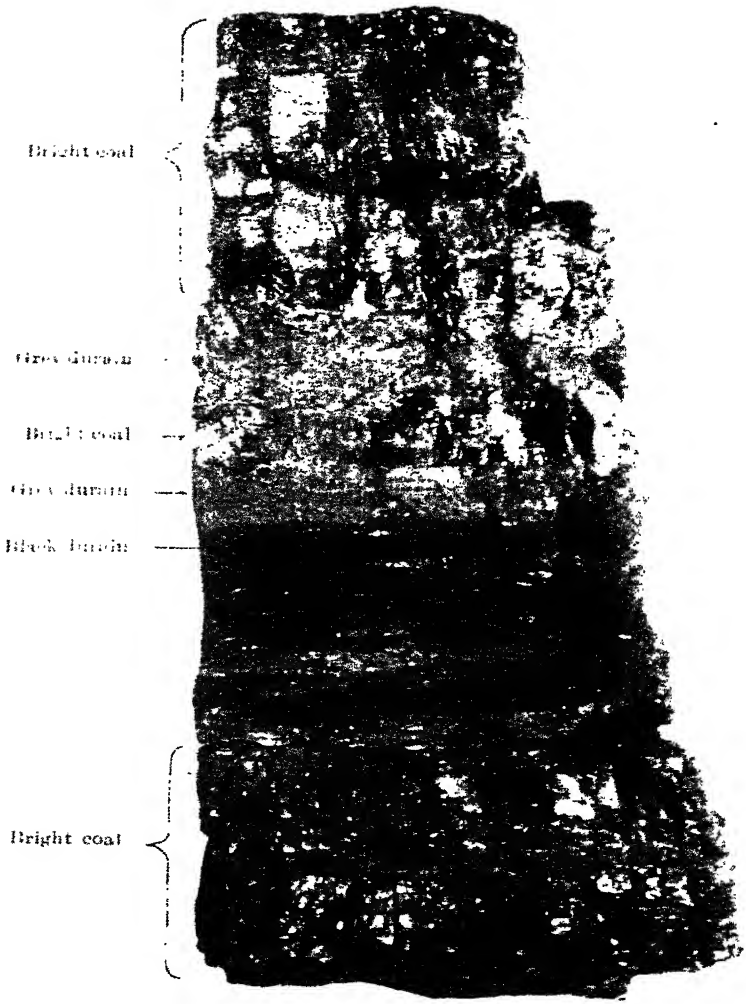


FIG. 70.—Photograph of Grey and Black Durain in Upper Beeston Coal (Wandless and Macrea).

seams of the Yorkshire coalfield, and the other of which the hydrogen-content is higher than in the associated clarain. An explanation of this has been given by A. M. Wandless and J. C. Macrea (*Fuel in Science and Practice*, 1934, **13**, 4), who have distinguished in the Upper Beeston seam (West Yorkshire) a "grey" and a "black" durain of different compositions, as shown in Table LXXIX.

TABLE LXXIX.—ANALYSES OF GREY AND BLACK DURAIN IN UPPER BEESTON SEAM.

	Moisture (Per cent., air-dried coal)	Ash	Carbon (Per cent., Parr's basis)	Hydrogen	Vol. Matter
Grey durain . .	4·2	4·3	86·7	5·07	34·5
Black durain . .	3·5	4·8	85·9	6·12	51·1
Clarain . .	5·3	2·9	84·7	5·56	39·4
Vitrain . .	6·6	1·5	85·4	5·22	35·0

The grey durain has a much lower and the black durain a much higher hydrogen-content than the clarain and vitrain with which they are associated in the Upper Beeston seam. A section of the Upper Beeston seam, indicating the grey and black durain bands, is reproduced in Fig. 70.

Although durain is usually a minor constituent of the commercial grades of coal used for coke-making, it may, on occasion, equal the clarain in amount, and it then modifies considerably the coking properties of the coal. Fig. 71 illustrates the effect of progressive decrease in its swelling-power obtained on adding different proportions of durain to the bright coal from the Barnsley seams from a number of collieries.

A durain by itself usually does not swell in the Sheffield Laboratory Coking Test. Individual lumps of durain may swell to a small extent on heating, usually less than 30 per cent., but when the durain is of low rank or of low hydrogen-content even individual lumps shrink on heating. The effect on its swelling-power of blending a bright coal with durain is illustrated in Fig. 72, which shows the results of "capsule" tests (see *Fuel in Science and Practice* 1934, **13**, 356) on durain from the Parkgate seam alone, and when blended with 5, 10, 20, 30, 50, 70 and 90 per cent. of bright coal from the same seam. When the blend contains less than 70 per cent. of durain the shapes of the individual particles are no longer evident in the cokes.

Provided that it is not present in excess, and is uniformly distributed, durain is a valuable constituent of a coking slack. The addition of 20 per cent. of durain to bright coal has been shown to raise the 1½-in. shatter index of the coke by from 5 to 10 points, the effect being most marked with coals of low rank. In Yorkshire the durain-content of small nuts may be under 20 per cent., and

that of coking slacks is usually from 20 to 30 per cent. When coking Midland coals, which are apt to contain fluctuating amounts of durain, the variations in durain-content should be recorded, the determination being conveniently made by the method described in the appendix to this chapter.

Vitrain.—The proportion of vitrain in a coal seam rarely exceeds 10 per cent. Occasionally, as in the Hutton seam, fairly broad bands of vitrain are encountered, but usually the bands are but a fraction of an inch in breadth. Vitrain is often considered to be the most valuable constituent of a seam of coking coal, but it is doubtful whether this view can be upheld. Vitrain often contains more inherent moisture than the accompanying clarain, and its hydrogen-content (and therefore its swelling-power) is usually lower, as is seen by the analyses in Table LXXX.

TABLE LXXX.—ANALYSES OF VITRAIN AND CLARAIN IN YORKSHIRE COKING COALS.

Seam		Analysis (per cent. on dry, ash- and sulphur -free basis)	
		Carbon	Hydrogen
Barnsley	Vitrain .	84·2	5·19
	Clarain .	83·9	5·39
Parkgate	Vitrain .	85·1	5·28
	Clarain .	85·6	5·38
Silkstone	Vitrain .	86·7	5·44
	Clarain .	86·9	5·60
Upper Beeston	Vitrain .	85·4	5·22
	Clarain .	84·8	5·54

Fusain.—Although fusain forms but a small part of a seam, it has an important influence on the coking of coal, as was shown in our first report ("Coke for Blast Furnaces," p. 186), and is discussed elsewhere in the present report.

The composition of fusain bears no apparent relationship to that of the coal in which it occurs. Published analyses are often unreliable because the fusain has not been entirely freed from coal, but a number, from different sources, have been collected in Table LXXXI, together with those of the corresponding bright coals.

The values for the Barnsley, Parkgate and Silkstone seams are mean values for from 12 to 16 analyses of seam sections in the Fuel Research Board's Reports, excluding those where the sulphur-content of the fusain exceeded 3½ per cent.

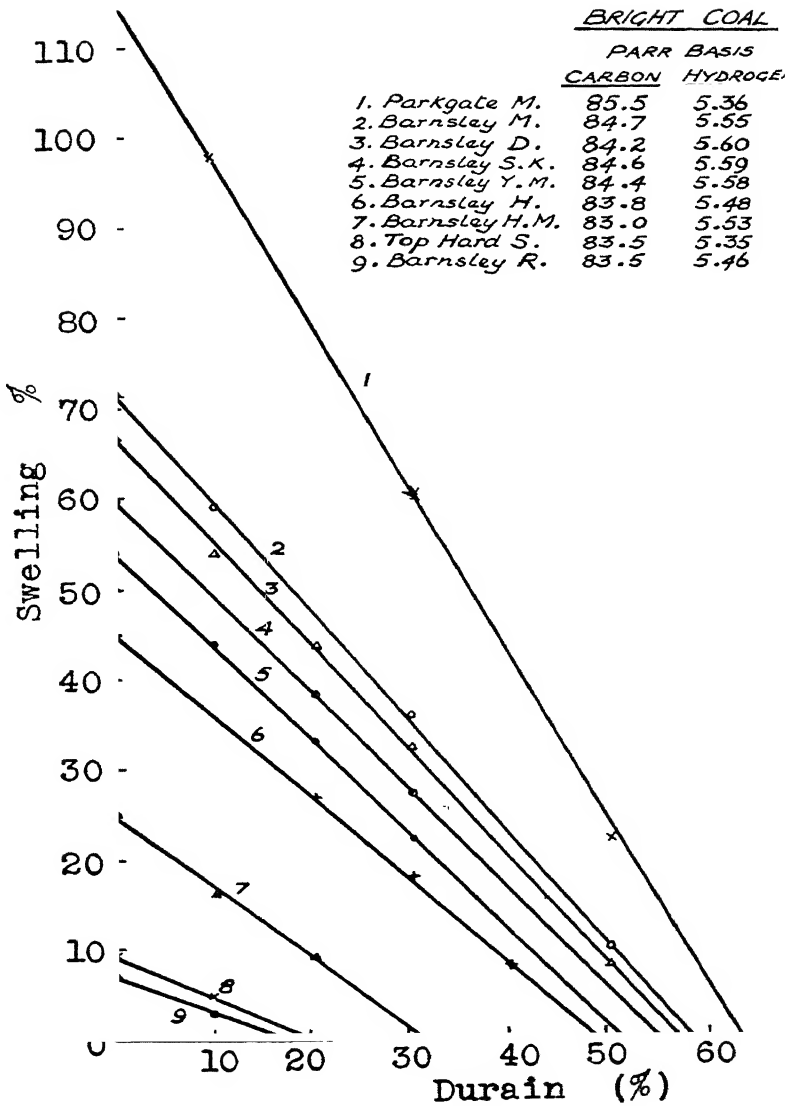


FIG. 71.—The Effect on the Swelling Power of Bright Coal of Adding Varying Amounts of Durain from the Same Seam.

TABLE LXXXI.—ANALYSES OF FUSAIN AND BRIGHT COAL.

Seam	Fusain					Bright Coal		
	Moisture.*	Ash *	Vol. Mat.†	Carbon ‡	Hydrogen ‡	Vol. Mat.†	Carbon ‡	Hydrogen ‡
Robins (S. Staffs.)	2·2	15·2	17·0	91·2	3·10	39·8	77·3	5·07
Deep Soft (Derby)	2·6	6·4	17·9	90·4	3·02	40·9	78·1	5·06
Dysart (Fife)	1·9	9·3	—	91·3	2·82	35·9	78·8	4·61
Barnsley (S. Yorks.)	2·1	15·2	20·6	94·6	2·26	36·6	80·8	4·86
Ell (Lanark.)	1·6	2·0	14·0	92·7	2·87	39·7	80·8	5·18
Top Slipper (S. Staffs.)	2·9	8·5	17·7	89·9	3·28	37·7	81·0	4·85
Deep (S. Staffs.)	1·9	5·1	13·7	91·0	2·84	39·6	81·6	5·46
Barnsley (S. Yorks.)	—	—	—	89·6	3·42	38·7	83·9	5·39
Beaumont (Northumb.)	1·6	9·9	29·9	90·1	3·72	37·5	85·2	5·34
Parkgate (S. Yorks.)	—	—	—	91·0	3·55	37·4	85·6	5·38
Ravine (Lancs.)	2·7	8·8	18·1	94·5	3·45	38·9	86·4	5·45
Arley (Lancs.)	1·4	4·7	12·6	90·8	3·47	34·9	86·4	5·78
Silkstone (S. Yorks.)	—	—	—	91·2	3·68	36·5	86·9	5·60
Hutton (Durham)	—	16·5	—	95·0	3·45	—	87·1	5·47
Three Quarters (Durham)	0·4	3·0	11·9	94·9	3·03	34·2	88·2	5·30
Brockwell (Durham)	0·5	8·7	12·4	93·9	3·05	27·4	88·4	5·10
Busty (Durham)	—	5·1	—	93·2	3·62	—	89·6	4·98
Brockwell A (Durham)	0·4	9·1	14·9	93·6	3·06	27·1	89·4	4·86

* Per cent., on air-dried basis.
† Per cent., on dry, ash-free basis.
‡ Per cent., on dry, ash- and sulphur-free basis.

An average value for the analysis of fusain lies between carbon 91 to 95 per cent., and hydrogen 2·3 to 3·5 per cent. A method of separating fusain, and of determining the quantity present in a slack or a dust, is described in the appendix to this chapter.

The general effect of the presence of a small proportion of fusain in a coking slack is to improve the quality of the coke, unless the coal is of low rank when its presence may tend to make the coke abradable.

It has been shown that the swelling-power of bright coal can be related fairly closely with its ultimate analysis, and that the addition of durain to a bright coal reduces its swelling-power in proportion to the amount added. The durains used for the tests upon which this observation is based varied in hydrogen-content (as recorded in Table LXXXII), but there is no indication that the variations were of any moment. The curves relating the swelling-power of the blends of bright coal and durain with the amounts of durain present (Fig. 71) lie nearly parallel whether the durains contained 4·95 or 5·30 per cent. of hydrogen. The comparative effect of durain and fusain in reducing the swelling-power of bright coal is illustrated in Fig. 73, which shows that fusain is about four times the more effective.

The swelling-power of a blend of durain and bright coal is less than the hydrogen-content of the blend would lead one to expect;

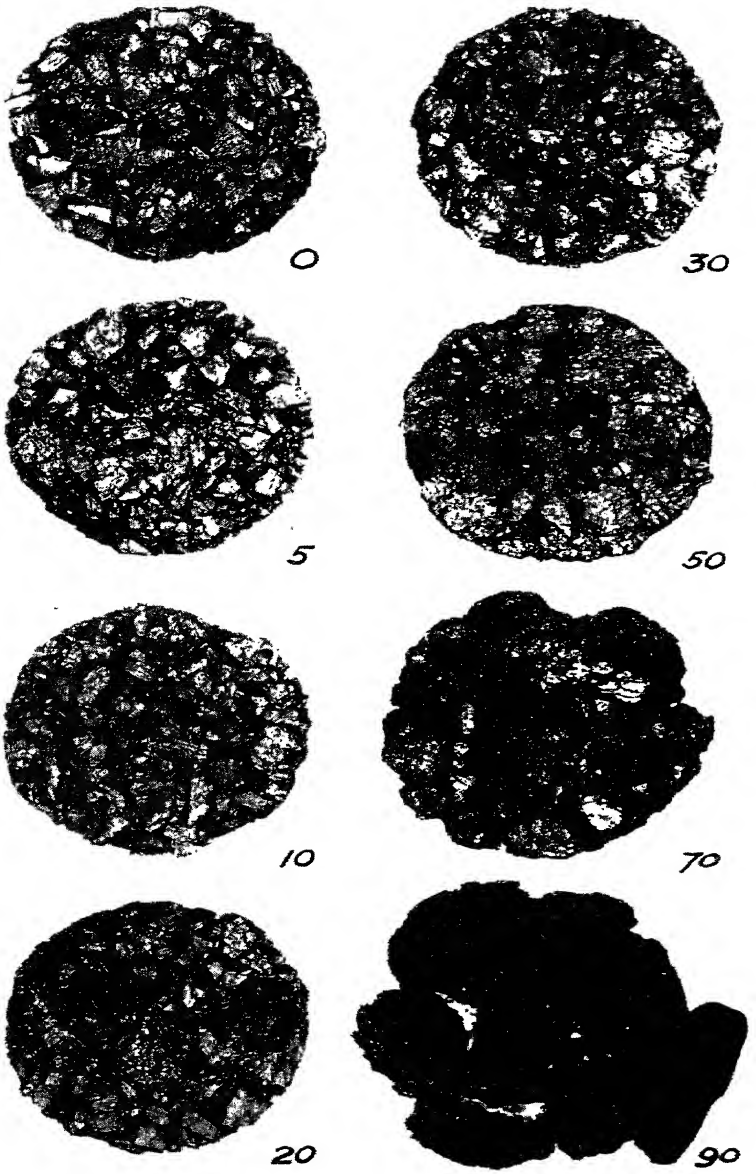


FIG. 72.—The Effect of Blending Bright Coal and Durain in Different Proportions on the Appearance of Coke made in a Capsule Test. (Figures are per cent. of bright coal).

that is to say, it is less than for an unblended bright coal of the same hydrogen-content. Similarly, the effect of the blending of fusain with a bright coal on its swelling-power is out of proportion to the reduction in hydrogen-content that it causes. The physical properties of the durain and, more markedly, of the fusain would therefore seem to be important in this respect.

TABLE LXXXII.—ANALYSES OF SLACKS FROM THE BARNSLEY SEAM AND OF THE DURAIN AND BRIGHT COAL IN THEM.

Slack No.	Slack		Durain		Bright Coal		Durain in Slack per cent.	Shatter Indices of Coke from Slack	
	Carbon *	Hydrogen *	Carbon *	Hydrogen *	Carbon *	Hydrogen *		1½-in.	½-in.
I .	85·6	5·40	85·8	4·95	84·2	5·60	47	79	96·9
VIII .	85·2	5·37	85·6	5·18	84·4	5·58	18	81	97·5
VII .	84·7	5·50	85·3	5·27	83·8	5·48	20	82	97·4
VI .	84·5	5·43	86·0	5·30	84·6	5·59	27	84	97·5
V .	83·6	5·56	84·1	5·23	83·0	5·53	40	76	97·2

* Per cent. on Parr's unit coal basis.

The Microstructure of Banded Bituminous Coal.—M. C. Stopes has recently amplified her original classification of banded bituminous coals, which was based on macroscopical examination, to include microscopical data (see *Fuel in Science and Practice*, 1935, **14**, 6) and an international conference held at Heerlen, Holland, in September, 1935, has agreed to the following system for international use (*Fuel in Science and Practice*, 1936, **15**, 14), the term "rock type" being applied to the macroscopic unit and the term "maceral" to the microscopic unit :

Rock Types.	*Macerals.*
Fusite (Fusain) .	Fusinite
Vitrite (Vitrain) .	Semi-fusinite Vitrinite
Clarite (Clarain)	Vitrinite { Collinite, Tellinite } Exinite
Durite (Durain)	Micrinite (Opaque Substance) Exinite Resinite

Semi-fusinite is intermediate in character between fusain and vitrain. Exinite includes all the recognisable plant entities of "rational" analysis that are of high hydrogen-content (but those of low hydrogen-content would include fusinite). Micrinite, which

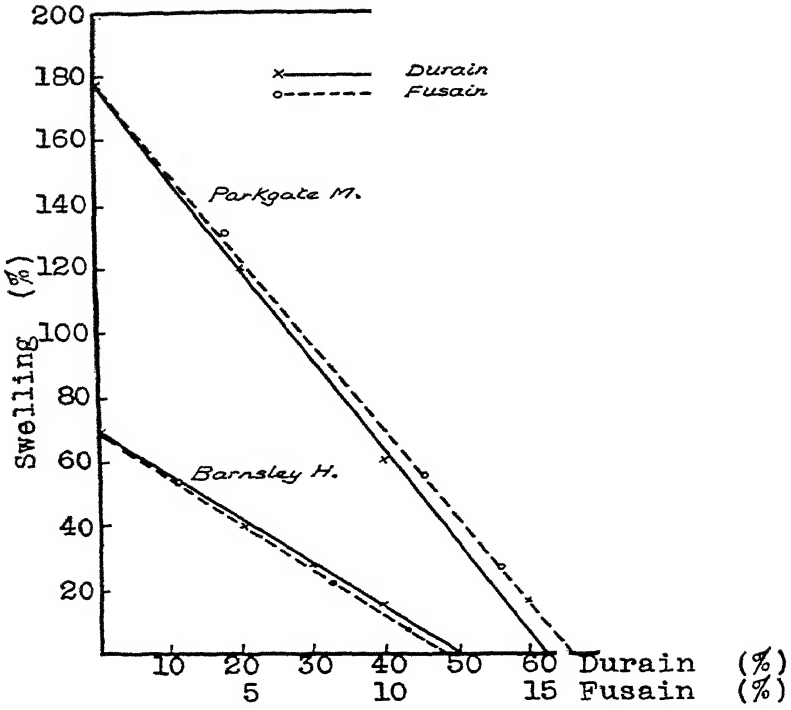


FIG. 73.—The Comparative Effect on Swelling Power of Bright Coal of Adding Durain or Fusain.

may occur in durain in considerable quantity, is of low hydrogen-content and may resemble fusinite in properties.

The principal constituent of coking slacks, clarain, can be regarded as containing up to 25 per cent. of exinite and fusinite, the remainder being vitrinite.

APPENDIX

THE RECOVERY OF FUSAIN FOR EXPERIMENTAL PURPOSES

Fusain often occurs, for example, in the Parkgate seam, South Yorkshire, in large lenticular pieces, and for experimental purposes can be collected from the picking belts instead of being thrown to waste. The crude fusain, mixed with a certain proportion of bright coal and pyrites, is tapped lightly on a 10-mesh I.M.M. screen. The most impure oversize is discarded and the rest, mixed with bright coal, is treated in a small rod mill with 5 bars during from 5 to 10 minutes and re-screened on 10 mesh, the oversize being discarded. The yield is about 30 per cent. and a typical analysis is as follows:

(1) Bright Coal, 60–100 m. (×32).

(2) Bright Coal and Fusain, 60–100 m. (×32).

(3) Bright Coal, through 200 m. (×40).

(4) Bright Coal 50%, Fusain 50%, through 200 m. (×40).

Size, mesh I.M.M.	Weight, per cent.	Fusain, per cent.
10–60	48	80
60–100	2	75
100–200	8	85
through 200	42	95
	100·0	86·5

The amounts of fusain in the different sizes were recorded by an experienced microcopist who made an estimate of particles which had the typical outline of fusain illustrated in Fig. 74. Such counts have been checked by a chemical method of estimation which is based on the fact that fusain is less readily attacked by oxidizing agents than are the other banded ingredients of coal dust. The method has been described by F. Heathcoat (*Fuel in Science and Practice*, 1930, **9**, 452) and has been improved by R. Belcher (*Fuel in Science and Practice*, 1935, **14**, 181) and can be outlined as follows :

THE ESTIMATION OF FUSAIN.—Grind the dried sample to pass a 200 I.M.M. mesh sieve. Boil three 1-gramme samples during seven hours in boiling-flasks fitted with ground-in reflux condensers with the following strengths of Schultz solutions :

(*a*) 60 cc. 4N HNO_3, 3 grammes $KClO_3$, 40 cc. water.
(*b*) 80 cc. 4N HNO_3, 3 grammes $KClO_3$, 20 cc. water.
(*c*) 100 cc. 4N HNO_3, 3 grammes $KClO_3$.

Filter each sample through a tared sintered-glass crucible (Jena 2G3) and wash once with water. Transfer the residues into 600 cc. beakers, add 40 cc. N 1 NaOH and 200 cc. of water and boil gently during 30 minutes. Add water to make the total bulk nearly 600 cc. and allow to stand overnight. Remove the supernatant layer by means of a sintered-glass filter stick (Jena 9G3). Should any particles adhere to the glass plate, introduce water inside the stick and apply gentle pressure by means of the mouth. Filter the residues through the corresponding crucibles, wash with hot water, followed by dilute hydrochloric acid, and finally with hot water. Dry during one hour in an air-oven at 105° C. and weigh. Determinations of ash on the original sample and the residue are necessary to correct the estimations to an ash-free basis.

The employment of oxidising solutions of different strengths is to determine whether gradually increasing oxidation decreases materially the amount of residue obtained. Although each result is rather low, owing to partial oxidation of the fusain, the use of solutions of graduated strengths has not so far given significant differences. Work is still proceeding on this test, but it seems likely that the use of only the strongest oxidising solution should suffice.

DETERMINATION OF FUSAIN BY A MICROSCOPIC METHOD.—Fusain is concentrated in the screen sizes of dusts below 60-mesh I.M.M., and it usually suffices to obtain screen fractions of 60 to 100, 100 to 200, and through 200 mesh, with a statement of the percentage of these fractions in the total dust. A dust of 60 to 100 mesh size is viewed by oblique light (e.g. from a ring of small lamps on the stage of the microscope), to reveal the typical pattern of the surface of fusain particles (Fig. 74, Nos. 1 and 2) with some light transmitted downwards by reflection to reveal the outline of the particles, the magnification being say, × 32. A dust of 100 to 200 mesh size is viewed by transmitted light (magnification say × 100) to reveal the outline and a vertical light is used for confirmation (magnification, say, × 300) to reveal the pattern on the surface, or the difference in reflective power of fusain and other coal particles. A dust through 200 mesh size is mounted in a spot of cedar-wood oil and spread with a cover glass to a single layer, being viewed by transmitted light to reveal the outline (Fig. 74, Nos. 2 and 3). The magnification used is × 100. A dust which has not been air-borne may reveal the cellular structure shown in Fig. 74, No. 6, but in a dust separated at a dedusting plant the difference in outline is the chief distinctive feature. A count is made of the coarser dusts, but an estimate of the percentage of fusain particles must suffice for the finer dusts.

THE DETERMINATION OF THE DURAIN CONTENT OF A COKING COAL.—The method depends on the fact (illustrated in Fig. 71) that the progressive addition of durain to bright coal gives a progressive reduction in its swelling power. It is necessary to pick, from a coking slack, pieces of pure bright coal and of pure durain, to determine the swelling power of the bright coal and of bright coal with suitable mixtures, say, 10, 20 and 30 per cent. of durain, in the Sheffield Laboratory Coking Test, and to graph the results. The swelling power of the coking slack is then determined and from the graph the percentage of durain is read off.

Since the results could be affected by the presence of fusain, it is necessary to use coal of 5 to 20 mesh size to enable fusain to be removed with the undersize. In Yorkshire coals this precaution has usually been successful, but pieces of hard fusain may still be found in the picked bright coal. It is, of course, necessary to pick a large quantity of coal to obtain a representative sample of bright coal and durain. Some difficulty may be experienced with fine coking slacks since the picking of bright coal is easier in comparatively coarse lumps. It is usually necessary to wash a coking slack to remove the dust before picking. At one colliery, where the coals were examined in greater detail, the bright coal picked from slack, singles, doubles, cubes and large coals was of the same swelling power and composition (Table CLVX) and it may, therefore, be satisfactory, as it is simpler, to pick pure bright coal from a larger nut size of coal.

CHAPTER XIV

THE DECOMPOSITION OF COAL

WHEN considering the composition of coals, it was shown that the coals of Great Britain, in general, range in carbon-content between about 78 and 95 per cent. (on Parr's "unit coal" basis), those of highest carbon-content being the anthracites, the hydrogen-content of which may be as low as 3·5 per cent.; whilst those which can be used, by themselves, for coke-making lie within a range of carbon-content of 82·5 to 91 per cent., and contain more than 4·6 per cent. of hydrogen. Throughout the whole range of natural fuels—peats, brown coals, lignites, bituminous coals and anthracites—the gradual change of rank has involved an elimination of water, carbon dioxide and methane from the "ulmins" which constitute the major proportion of all of them irrespective of their rank.

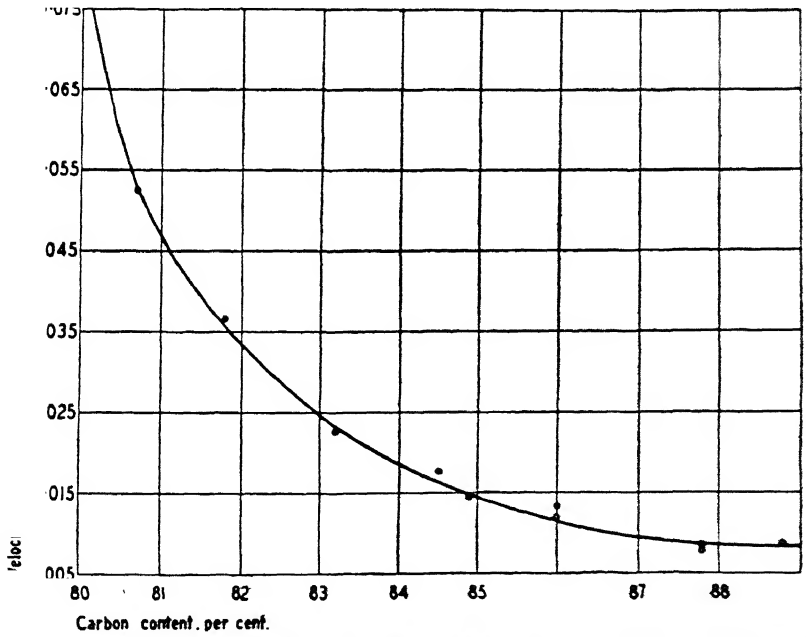


FIG. 75.—The Reactivity to Permanganate of Coals of Varying Carbon Content. (Francis.)

Although but little is known of the molecular structure of the ulmins, such properties as their chemical reactivity and their ease of decomposition have been studied in several ways. The reactivity of the ulmins of a number of coals of different rank towards a mild oxidising agent was recorded in our first report ("Coke for Blast Furnaces," p. 167), where it was shown that the reactivity decreases as the rank of the coal increases. In Fig. 75 the reactivity of the ulmins is plotted against the carbon-content of the coals (dry, ash-free basis). The reactivity was determined on 0·5 gramme of coal of size through 120 and on 150 mesh (I.M.M. standard), using an alkaline solution of potassium permanganate of normal (about 3 per cent.) strength, and is expressed as the velocity constant of the reaction after 50 minutes (*see* W. Francis, *Fuel in Science and Practice*, 1933, **12**, 133). The reactivity increases rapidly as the carbon-content of the coal decreases below 83 per cent., which is near the lower limit of carbon-content for a coal to possess swelling-power in the Sheffield Laboratory Coking Test.

In Fig. 76, for a series of coals, the reactivity of residues obtained after heating the coals to different temperatures are recorded, using alkaline potassium permanganate of normal strength on material of size through 60 and on 120 mesh (I.M.M. standard). The coals (of through 60-mesh size) were heated to the specified temperature in the apparatus used for the Sheffield Laboratory Coking Test (silica retort) at a rate of rise of temperature of 1° C. per minute. Analyses and other data for the coals used are recorded in Table LXXXIII.

TABLE LXXXIII.—THE REACTIVITY OF HEATED COALS. ANALYTICAL AND TEST DATA FOR THE COALS USED.

Name of Coal	Moisture per cent. air dry	Volatile Matter Per cent.	Carbon (on Parr's basis)	Hydrogen	Sheffield Laboratory Coking Test: Softening Temp. °C.	Temp. of Initial Expansion °C.	Temp. of Final Expansion °C.	Per cent. Expansion
Ryder (S. Staffs.)	6·4	38·2	82·2	5·49	267	407*	—	nil
Barnsley (S. Yorks.)	2·1	36·7	84·4	5·60	351	390	414	63
Parkgate (S. Yorks.)	1·5	36·6	86·9	5·40	343	394	412	126
Two Foot Nine (S. Wales)	0·5	21·9	89·7	4·65	403	444	464	94

* Final contraction.

The results given in Fig. 76 show changes in the reactivity after heating which, for the three coking coals which swell, begin near the softening-point and, for two of them, attain a maximum at the temperature of initial expansion. With the coal of highest rank (Two Foot Nine), however, the residue after heating to a temperature higher than that of initial expansion has the maximum reactivity.

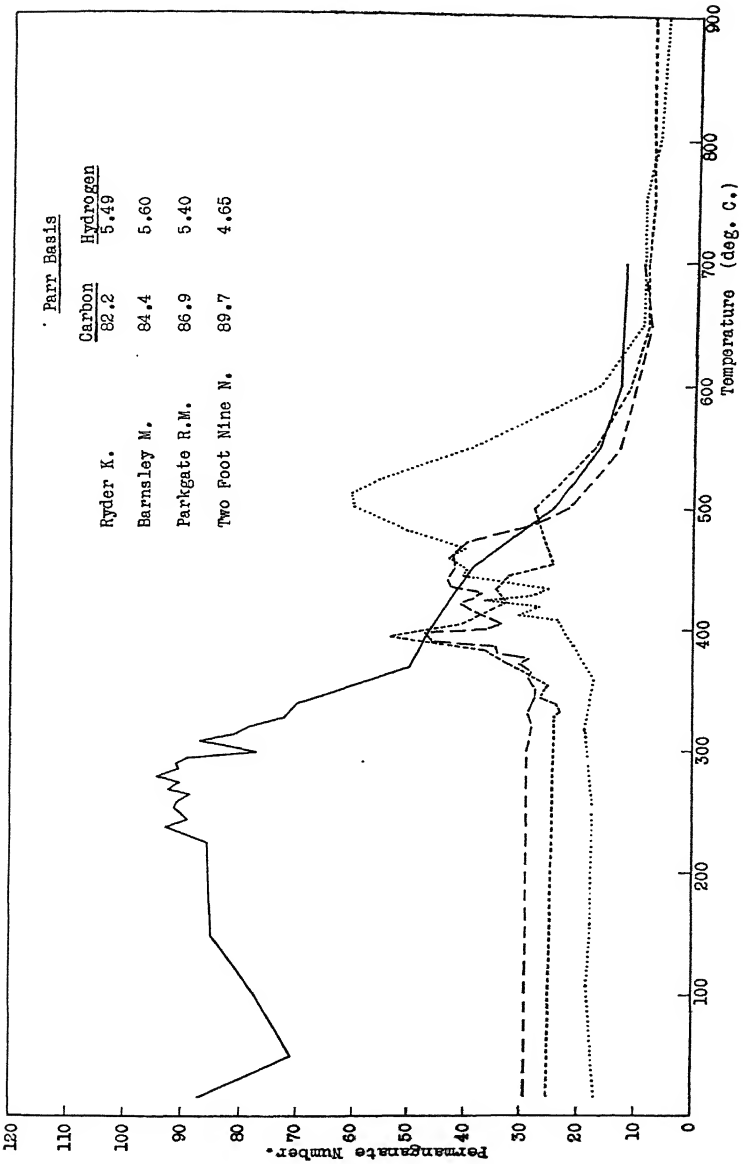


FIG. 76.—Permanganate Numbers of Coals Heated to Different Temperatures.

Other experiments have shown that the plasticity of a charge of coal heated under the conditions of the Sheffield Laboratory Coking Test (*i.e.* free to expand) ends at about 440° C. for the Parkgate, and at about 480° C. for the Two Foot Nine coal. With the latter coal heated under conditions such that expansion is prevented, plasticity does not end until about 520° C. (see *Fuel in Science and Practice*, 1933, **12**, 371). In general, it would appear that the reactivity of a charge of coal that has been heated begins to decrease if the heating is carried beyond the point at which plasticity ceases. The residues from each of the four coals referred to in Table LXXVIII and Fig. 76 had about the same reactivity when the heating was carried beyond 700° C.

It must be pointed out that the reactivity of coals and cokes as measured in these experiments is influenced considerably by the amount of surface exposed to the oxidant (W. Francis, *loc. cit.* p. 135). Although all the materials were carefully sized between 60 and 120 mesh, those obtained after heating beyond the temperatures at which plasticity began were porous, and the effective surfaces of the particles would thus be increased. The "peaks" of reactivity in the curves for the swelling coals (Fig. 76) might be due, in part, to this increase in surface. On the other hand, the major increase in porosity occurs after the temperature of initial expansion has been passed, whereas there has been a considerable increase in the reactivity of the residues of the swelling coals heated at the temperature of initial expansion. Some of the increase in reactivity of residues obtained on heating a coal beyond its softening-point is therefore due to chemical changes; most probably, as will be seen later, to changes arising from decomposition of the ulmins.

The Decomposition-Point of Coal.

The decomposition-point of coal has been studied in the Department of Fuel Technology of Sheffield University by two principal methods: by slowly distilling, under vacuum, 250-gramme charges of coal and collecting and analysing the gases and oils evolved; and by raising the temperature of a thin layer of powdered coal 0·5° C. per minute and noting the rate of evolution of gases.

The Decomposition of Coal by Slow Distillation.—A vertical retort of glass, holding 250 grammes of coal sized between 20 and 60 mesh, was supported with its mouth downwards within an electric furnace and connected through a system of cooled condensers with a vacuum-pump (*see* R. Holroyd and R. V. Wheeler, *Fuel in Science and Practice*, 1930, **9**, 40).

When carrying out a distillation, the charge of coal was first evacuated at room-temperature during 50 hours (to remove occluded gases), and its temperature then raised during 2 days to 100° C. at which it was maintained during 5 days. Thereafter, distillation

was continued during 5-day periods at 200°, 275°, 300°, 325°, 350° and 400° C., the whole test lasting about 7 weeks. Heavy oils were collected in an air-cooled receiver and light oils in a series of two receivers cooled by solid carbon dioxide dissolved in ether. Gases passed through the vacuum pump to holders.

It was found that, with bituminous coals of over 80 per cent. carbon-content, none of the constituents decomposed markedly below about 200° C., but between 220° and 300° C. the major part of the free hydrocarbons (the "γ_1" constituents obtained by solvent analysis) distilled off unchanged. At about 300° C. such plant remains as spore-exines and cuticles began slowly to decompose. Above 300° C. (within the range 300-375° C.), marked decomposition of the ulmins began.

The liquid products of decomposition of the plant remains are characterised by the presence of unsaturated hydrocarbons, and their gaseous products by the oxides of carbon. The decomposition of the ulmins is marked by a sudden increase in the volume of the gases evolved, which are rich in paraffin hydrocarbons, and by the appearance of phenolic and aromatic compounds in the oils. The temperature of decomposition of the plant remains does not vary with the rank of the coals, but the decomposition-point of the ulmins rises with their rank, as is shown in Table LXXXIV.

TABLE LXXXIV.—THE DECOMPOSITION-POINTS OF COALS (HOLROYD AND WHEELER).

Coal	Analysis (on "Pure Coal" Basis), per cent.*			Liquor from Dried Coal, Per cent.†	Phenols, per cent.†	Decomposition Point, ° C.
	Carbon	Hydrogen	Oxygen			
Seven Foot (S. Staffs.)	78·2	5·16	14·0	—	—	295
Thick Vitrain	78·3	5·15	14·5	6·0	2·2	297
(S. Staffs.) Durain	80·9	4·71	12·3	5.4	1·4	300
Barnsley Vitrain	81·2	5·31	11·2	4·1	0·9	310
(S. Yorks.) Durain	82·7	5·23	9·6	4·1	0·9	310
Wigan Six Foot (Lancs.)	82·8	5·54	9·1	4·1	1·9	317
Parkgate (S. Yorks.)	83·4	5·52	—	—	—	327
Silkstone (S. Yorks.)	83·6	5·38	7·9	1·8	0·8	327
Six Quarter (Cumberland)	86·6	5·08	6·3	2·3	1·1	327
Busty (Durham)	87·4	4·72	5·9	—	—	337
Busty (Durham)	88·4	5·27	4·2	—	—	342
Black Vein (S. Wales)	88·5	5·02	4·1	1·5	0·6	342
Two Foot Nine (S. Wales)	89·8	4·18	3·7	0·8	0·1	362

* *i.e.* corrected for mineral matter, moisture and pyritic sulphur.
† Up to 400° C.

In Table LXXXV, comparison is made of the changes in composition of the gases and oils evolved over different temperature ranges for the vitrain (V) and durain (D) of the Barnsley seam.

TABLE LXXXV.—CHANGES IN COMPOSITION OF GASES AND OILS WITH TEMPERATURE FOR BARNSLEY VITRAIN (V) AND DURAIN (D).

Temp. Range °C.	Gases: Volume, cc. per gramme		Gases: Paraffins, per cent.		Gases: CO_2, CO and H_2S, per cent.		Heavy Oils: Per cent. on coal		Heavy Oils: Phenols *		Heavy Oils: Unsaturated Hydro-carbons *		Heavy Oils: Aromatic Hydro-carbons *		Heavy Oils: Saturated Hydro-carbons *		Heavy Oils: Resins *	
	V.	D.	V.	D.	V.	D.	V.	D.	V.	D.	V.	D.	V.	D.	V.	D.	V.	D.
0–100	0·5	0·3	75	77	25	22	nil	nil										
100–200	0·3	0·3	26	30	73	70	nil	nil										9
200–280	0·5	0·3	24	19	70	76	0·15	0·23	nil	nil	35	30	20	17	25	27	nil	
280–300	0·7	1·2	46	39	42	52	0·25	0·65	nil	6		36		12		9		8
300–310	1·4	3·1	62	56	24	34	0·45	3·36	18	6	tr.	37	22	8	16	6	13	12
300–320	2·3	3·4	68	64	20	22	0·58	4·43	21	4	5	38	19	8	10	5	10	14
320–330	4·1	3·3	73	73	15	13	0·73	1·47	22	7	8	23	12	11	4	7	19	13
330–340	5·7	3·6	79	77	13	11	0·65	0·90	22	7	11	27	5	13	3	7	30	17
340–350	6·2	4·2	82	76	11	12	0·37	0·62	30	5	16	27	5	16	tr.	6	22	10
350–375	15·0	11·5	82	78	10	10	0·32	0·72	31	12	19	20	9	8	tr.	3	16	—
375–400	16·0	13·3	90	80	7	6	0·10	0·14	tr.	tr.	20	36	tr.	tr.	tr.	tr.	—	—
Total to 400	49·9	42·4	70	64	19	24	3·60	12·52	25	7	12	33	12	9	8	6	19	12

* Per cent. on total heavy oil.

Taking the Barnsley vitrain and durain as examples (the original work gives details for the other coals), the decomposition-point is indicated primarily by a marked increase in the evolution of gases at 295–300° C. for the vitrain, and at about 310° C. for the durain. The composition of the gases changes from being mainly the oxides of carbon before the decomposition-point of the ulmins to mainly the paraffin hydrocarbons afterwards. The degree of decomposition

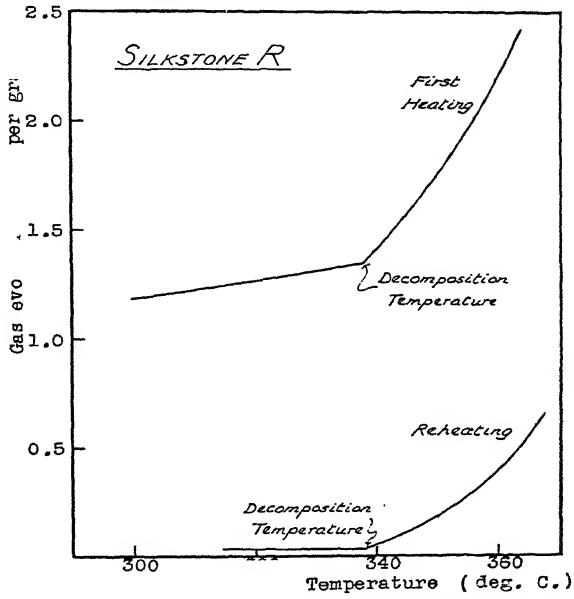


FIG. 77.—Graph illustrating Method of Determining the Decomposition Temperature of Coal (Hibbott and Wheeler).

of the ulmins at the outset is small, and it does not become extensive until the decomposition-point has been exceeded by about 50° C., when a fairly constant evolution of gases of about 6½ c.c. per gramme of coal for each rise of temperature of 10° C. is attained. At that stage, nearly all the oils have been evolved from the coal.

The proportions of the types of heavy oils evolved over the different temperature-ranges recorded in Table LXXXV show clearly the differences in character between the vitrain and the durain from the same seam of coal. The plant entities in the durain are responsible for its higher yields of oils over each range of temperature, oils in which unsaturated hydrocarbons preponderate.

The hydrocarbon oils which distil from a bituminous coal under vacuum below the temperature of decomposition of the ulmins have been found to correspond in quantity and character

with the "hydrocarbon" fraction (soluble in light petroleum) obtainable by solvent analysis; and the resinous oils distilled, though rather less in quantity, were similar in character to the "resins" (soluble in ethyl ether) so obtained.

The decomposition-point of a coal (*i.e.* of its ulmins) is an important property to determine. The gradual distillation in a vacuum, as used for the values recorded in Table LXXXIV, is obviously too laborious a method of determination for frequent use. H. W. Hibbott and R. V. Wheeler (*J. Chem. Soc.*, 1934, 1084) therefore devised a simpler method in which 1 gramme of the coal, of between 100 and 200 mesh size, is raised in temperature at a rate of 0·5° C. per minute and the gases evolved collected continuously. The coal is layer 3 mm. thick in the flat "bulb," measuring 5 by 2·5 by 1 cm., of a glass retort connected to a Sprengel mercury-pump through which the gases evolved as the coal is heated are withdrawn. On plotting the rate of evolution of gas against the temperature, a sudden increase is observed at the decomposition-point. As a precaution, to avoid the possible masking of the decomposition-point by gases (mainly oxides of carbon) evolved below it, a preliminary heating of the charge of coal for a short time can be carried 30 to 40° C. above the temperature at which a sudden increase in the rate of evolution of gas occurs; on then cooling the charge and reheating it, only traces of gas are evolved below the decomposition-point (see Fig. 77).

The decomposition-points of a number of coals as determined by Hibbott and Wheeler are recorded in Table LXXXVI, where they are compared with the values obtained by Holroyd and Wheeler with their much slower rate of heating.

TABLE LXXXVI.—THE DECOMPOSITION-POINTS OF COALS.

Coal	Carbon, per cent.	*Decomposition-point* ° C.		Difference ° C.
		Holroyd and Wheeler	Hibbott and Wheeler	
Seven Foot	78·2	295	312	17
Barnsley, vitrain	81·2	307	320	13
Barnsley, durain	82·7	310	324	14
Wigan Six Foot	82·8	317	333	15
Parkgate, clarain	83·4	327	337	10
Silkstone	83·7	327	337	10
Parkgate, run-of-mine	85·0	—	346	—
Virtuewell	86·3	—	350	—
Six-quarter	86·6	322	335	13
Busty	87·4	337	352	15
Busty	88·4	342	355	13
Pensford No. 2	88·4	—	360	—
Two Foot Nine	89·8	362	374	12

The values obtained for the decomposition-points by the "rapid" method of determination are from 10 to 15° C. higher than those obtained when the rate of heating was very slow. The effect of altering the rate of heating in their determinations was shown by Hibbott and Wheeler as follows:

Rate of Heating ° C. per minute	Decomposition-point °C.	
	Pensford	Busty
1·0	372	365
0·8	366	359
0·5	360	352
0·3	355	346
Nil (extrapolated)	348	337

If the rates of heating are plotted against the decomposition-points, the values lie on straight lines which, by extrapolation for an infinitely slow rate of heating, give for the absolute decomposition-point of the Pensford coal 348° C. and for the Busty 337° C., the values obtained by Holroyd and Wheeler.

The Decomposition of Coal at a Rate of Heating of 1° C. per minute.—When, as in coking practice, the rate of heating a coal is faster than in the experimental work just described, the reactions which take place at a given temperature are incomplete. The decomposition-point may therefore appear to be raised above the absolute value by an amount dependent upon the rate of heating.

In experiments carried out by Allinson and Mott (*Fuel in Science and Practice*, 1933, **12**, 258), in which coals were raised in temperature in the Gray-King assay apparatus at a rate of 1° C. per minute, extensive decomposition of the coals was marked by the rapid production of gases, rich in hydrocarbons. This extensive decomposition occurred at about 360–380° C. for all the coals tested, and, for most of them, there was a second rapid increase in the rate of evolution of gas at about 380–420° C. (see Figs. 78 and 79). Except with coals of low rank, there was a rapid increase in the collection of oils at about the same temperature as for rapid evolution of gases. The reason for this close connexion between the apparent production of oils and the evolution of gas was traced to the fact that, below the temperature of sudden evolution of gas, oil liberated from the coal was adsorbed by the charge. The amount so adsorbed was as much as 6 per cent. (of the coal) for a coal of high rank.

Adsorption of oils by the charge to an appreciable extent was observed at the lowest temperature used, about 350° C., and probably would occur at lower temperatures. As the temperature was increased up to about 400–420° C., the amount of adsorbed

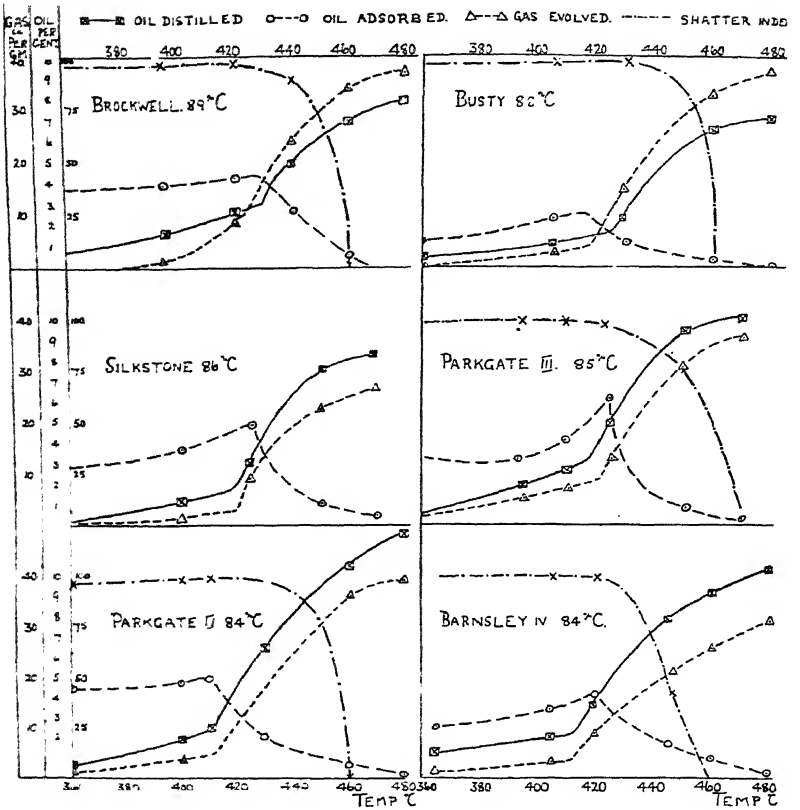


FIG. 78.—Evolution of Gas, Adsorption and Distillation of Oil from Coals of 89 to 84 per cent. Carbon Content on Heating to Different Temperatures (T° C.) at a Rate of 1° C. per min. The Effect of Residual Oil on the Shatter Index of Coke made on Heating Coal Residues at T° to 600° C.

oils increased to a maximum (*see* Figs. 78 and 79) and then decreased, when the amount of oils distilled increased. Apparently, the adsorptive-power of the charge of heated coal for oils was overcome when the rate of evolution of gas attained a critical value. The oils are retained as films on the surfaces of the particles of coal, but the rapid evolution of gas beneath the films can overcome the molecular forces of adhesion and thus entrain the oils. The evolution of gas at which the breaks in the curves in Figs. 78 and 79 occurred was usually about 4 c.c. per gramme of coal.

The production of gas from coal at two selected temperatures according to the method of heating is compared in Table LXXXVII, the quantities of gas being given in c.c. per gramme of coal.

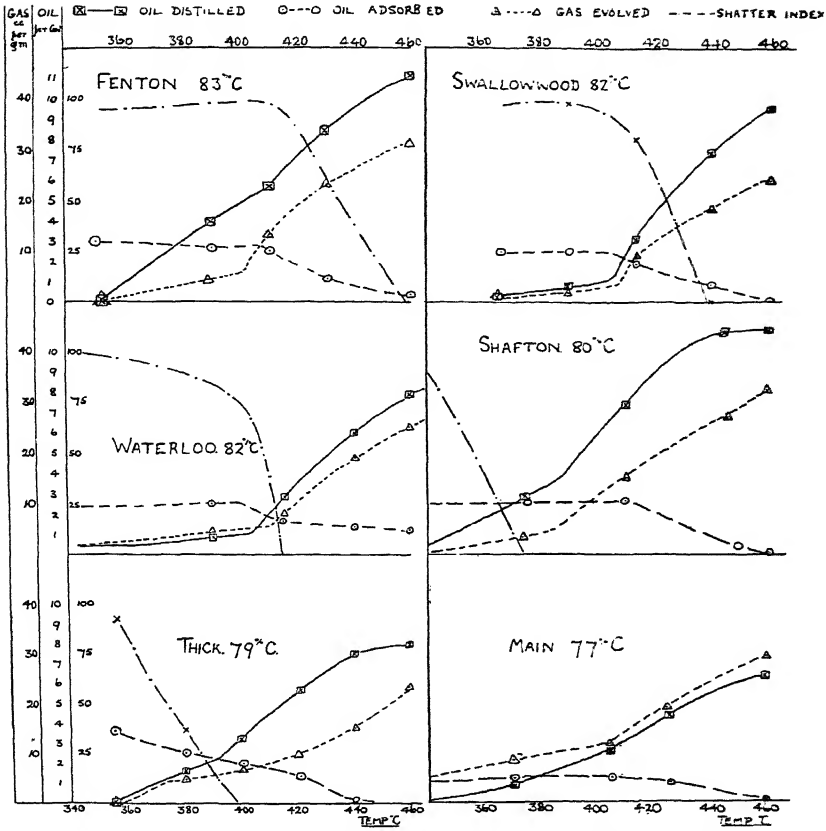


FIG. 79.—Evolution of Gas, Adsorption and Distillation of Oil from Coals of 83 to 77 per cent. Carbon Content on Heating to Different Temperatures (T° C.) at a Rate of 1° C. per min. Effect of Residual Oil on Shatter Index of Coke made on Heating Residues at T° C. to 600° C.

The volume of gas produced from coal at 350° C. is shown by the results given in Table LXXXVII to be considerably greater when that temperature is reached very slowly than when the rate of rise of temperature is 1° C. per minute. At the latter rate of heating, it is found that a temperature of about 420° C. must be reached before the amount of gas evolved equals that obtained at 350° C. by slow heating. It can be assumed that, under the conditions of heating adopted by Allinson and Mott in their work, extensive production of oil and gas from a coal would not occur until the temperature exceeded the "decomposition point" by about 75° C.

TABLE LXXXVII.—COMPARISON OF THE PRODUCTION OF GAS FROM COAL AT DIFFERENT RATES OF HEATING.

Carbon content of coal	1° C. per minute (Allinson and Mott)		0·5° C. per minute (Hibbott & Wheeler)	"Nil" (Holroyd & Wheeler)	
	350°	400°	350°	350°	400°
89	—	—	2	3	27
89	nil	2	—	—	—
88	nil	2·5	2	7	—
86	nil	1	3	14	—
85	nil	5	4·5	—	—
84	0·3	4	—	—	—
84	0·7	3	2	4	—
83	nil	5	5	14	—
83	—	—	—	18	—
82	0·7	3	—	—	—
81	0·5	5	6	22	53
80	0·2	5	—	—	—
78	—	—	—	18	50
Mean	0·2	3·5	3·4	12·5	43·5

Allinson and Mott's results (*see* Figs. 78 and 79) show that the degree of adsorption of oils by a coal during its heating varies with the rank of the coal. The curves in Fig. 80 relate the distillation of oil with the temperature of heating for a number of coals and show that the distillation of oil varies approximately with the rank of the coal. There are two groups of curves, the upper group comprising the three coals of lowest carbon-content. The curves for the coals of highest carbon-content (86 to 89 per cent.) are convex to the abscissa, whilst the curves for the upper group (non-coking coals) are concave.

Fig. 81 shows curves for the evolution of gas from the coals. The curves are crowded together more closely than in Fig. 80, but can still be divided into groups. The upper group includes Nos. 7, 10 and 12 (as in Fig. 80), but coal No. 11, which is in the upper group for the distillation of oil, is at the bottom of the lower group for the evolution of gas, and the curves for two coals of high carbon-content (Nos. 1 and 2) appear near the top of the lower group.

Since for each coal the evolution of gas and the distillation of oil are related (see Figs. 78 and 79), it is probable that the unusually high rate of distillation of oil from coal No. 7 is due to its unusually high rate of evolution of gas over the range 420–440° C., whilst it may be noted that with this coal, as with the coals of lowest rank, there is no pronounced break in the curves either for the evolution of gas or the distillation of oil. Since there is no direct relationship between the evolution of gas and the rank of the coal, it is evident that the relationship indicated (by Fig. 80) between the rank of a coal and its distillation of oil is dependent on the adsorption of oil.

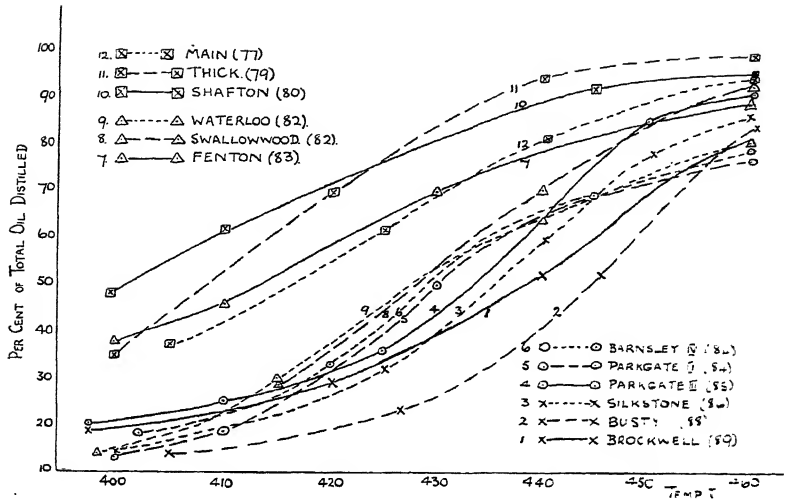


FIG. 80.—Per cent. of Total Oil distilled from Columns of Particles of Coals of Different Carbon Content at Various Temperatures.

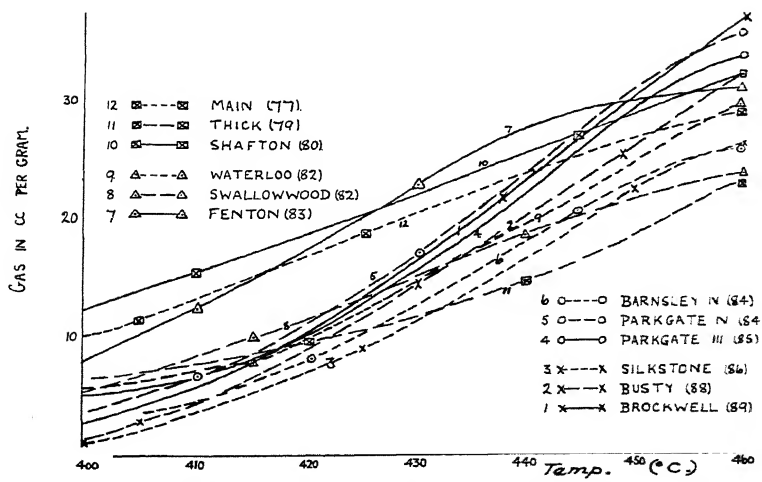


FIG. 81.—Total Evolution of Gas from Columns of Particles of Coals of Different Carbon Content at Various Temperatures.

The effect of the adsorption of oil during the heating of a coal has also been shown by comparing the behaviour of single lumps and crushed coals, as in Table LXXXVIII.

TABLE LXXXVIII.—THE EVOLUTION OF OIL FROM LUMP AND CRUSHED COAL. (Heated to the temperature of final expansion, or contraction, in the Sheffield Laboratory Coking Test. 400—440°C.).

Coal	Carbon-Content (on dry, ash-free basis)	Oil Distilled, per cent. of Total Evolved: From Lump Coal		Oil Distilled, per cent. of Total Evolved: From Crushed Coal	
Busty	89 to 84	–	Average 86%	26	Average 27%
Brockwell		87		28	
Parkgate III		–		25	
Silkstone		90		32	
Parkgate II		92		19	
Barnsley IV		25		33	
Fenton	83 to 77	–	Average 84%	46	Average 51%
Swallow Wood		71		28	
Waterloo		93		64	
Shafton		77		62	
Thick		–		70	
Main		97		37	

From the single lumps of the coals an average of 85 per cent. of the oil evolved at temperatures ranging from 400–440° C. was distilled, and there was no obvious relationship between the amount distilled and the rank of the coal. From the crushed coals, less than one-third (for good coking coals) or not much more than one-half (for poorly-coking or non-coking coals) of the oil evolved was distilled.

A comparison is made in Fig. 82 of the temperature-range over which oil was distilled from lumps and crushed coals, the results being shown as bands for coals of 82 to 86 per cent. carbon content. With the lumps there is a regular increase in the amount of oil distilled with increase of temperature and at about 440° C. the amount reaches 90 per cent. of the total evolved from the coal. With the crushed coals adsorption delays the distillation of oil until a temperature of about 420° C., and 90 per cent. is not distilled until about 490° C. is attained. Fig. 82 also shows separately the distillation of oil from several crushed coals, namely, two of high rank and three (non-coking) of low rank. The distillation of oils from the coals of high rank occurs less readily than from coals of 82 to 86 per cent. carbon-content, whilst from the crushed coals of low rank the oils are distilled more readily—almost as readily as from single lumps of such coals.

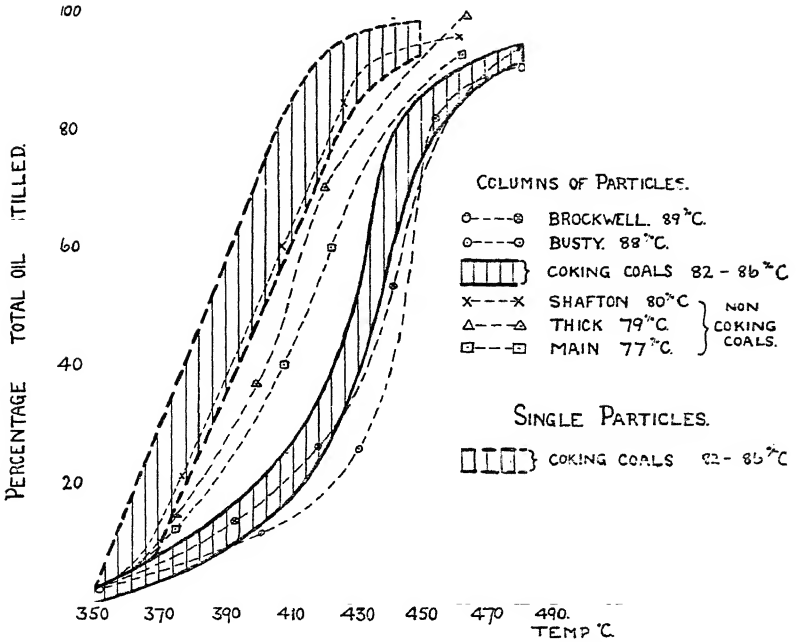


FIG. 82.—Comparison of Evolution of Oil from Columns of Particles and from Single Particles of Different Carbon Content.

The manner in which oils are released from a charge of coal during carbonisation is thus complex. From the difference in behaviour as between a single lump and a crushed charge of the same coal it is evident that, whereas oil may be released readily from a single lump, a charge may retain much of the oil by adsorption on the surface of the particles, and a high temperature, with a rapid evolution of gas, may be required to release it.

COKE-FORMATION AND THE RELEASE OF VOLATILE MATTER

Much attention has been paid by many investigators to relationships between the phenomena of coke-formation and the evolution of volatile matter. It is well known that coking coals of low volatile matter content (20 to 24 per cent.) yield the hardest metallurgical cokes, and it is believed that the expulsion of much volatile matter from a coal causes the numerous fractures (shrinkage-cracks), which render the coke from such a coal weak and frangible. As discussed in our first report (p. 179), these observations have led to the practice, where coals of low and high volatile matter content occur within reasonable distance, of blending the two types to produce a charge of average volatile matter content 20 to 24 per cent.

A favourite method of studying the behaviour of the volatile matter of a coal during coking has been to determine its loss continuously whilst the coal was being heated in a crucible suspended on a balance. E. Audibert (*Rev. de l'Ind. Min.*, 1926, **6**, 115; *Fuel in Science and Practice*, 1926, **5**, 229), for example, used such a method with a rate of heating of 1° C. per minute. Audibert drew a distinction between two classes of coals, one in which thermal decomposition may take place, without loss in weight, before there is any softening, and the other in which loss in weight precedes softening. It is doubtful whether such a distinction need be drawn. Most of the coals of Audibert's first class were of low and most of the second class were of high softening-point, and it is not easy to determine the softening-point with accuracy by the method Audibert used.

P. Damm (*Glückauf*, 1928, **64**, 1073; *Fuel in Science and Practice*, 1929, **8**, 163) was one of the first German workers to attempt to relate the evolution of volatile matter with the plastic phenomena during the heating of coal. He determined the "fusion-point" by noting when a pointed rod of iron, weighted to 500 grammes, began to sink through a briquette of coal rapidly heated to 300° C., and subsequently (at an unspecified rate) by stages of 10° C. He also measured the loss of volatile matter when the coal, contained in a small boat, was heated to various temperatures in a tube-furnace, the significant temperatures being below, during and above a range 25° C. below to 25° C. above the fusion-point (the "fusion-range"). Three coals which lost weight appreciably during the "fusion range" were found to have high swelling pressures, a fact which Damm considered confirmatory of Fischer's view (*Brenn. Chem.* 1925, **6**, 33; *Fuel in Science and Practice*, 1926, **5**, 466) that coals only swell if the temperature of decomposition of the "solid bitumen" they contain coincides with the temperature of fusion. Two coals which lost much volatile matter during the "fusion-range," and showed marked decomposition before that period, were non-swelling, which suggested to Damm that the whole of the coal, and not only the "solid bitumen," was concerned in its swelling. A weakness in Damm's study was that the determinations of fusion-point and of loss of volatile matter were not carried out under the same conditions of heating, and his arbitrary choice of a "fusion-range," 25° C. on either side of the fusion-point, does not seem to have had any justification.

W. Davidson (*Fuel in Science and Practice*, 1930, **9**, 489) heated charges of coal in small boats, at specified rates, and determined the loss in volatile matter at the beginning and at the end of the "plastic range" as measured by a modification of Foxwell's method, wherein the resistance of a column of crushed coal to the passage of a stream of nitrogen is measured whilst it is being raised in temperature (see *Fuel in Science and Practice*, 1924, **3**, 122). In each instance, the loss of volatile matter below the temperature assumed

TABLE LXXXIX.—THE LOSS OF VOLATILE MATTER FROM COAL DURING HEATING (DAVIDSON).

Coal	Moisture, per cent. air-dry	Vol. Matter (per cent. on dry ash-free basis)	Carbon	Hydrogen	Caking Index	Plastic Range ° C.		Volatile Matter and Plastic Range (see text)			Shatter Indices of Coke	
						Beginning	End	Before	During	After	1½-in.	½-in.
D.G. (Scotland	1·6	29·8	—	—	24	413	469	1·2	7·6	18·0	95	98·5
Victoria (Durham)	0·9	28·4	89·6	5·14	37	361	508	0·2	14·9	12·3	93	98·9
Brockwell (Durham)	1·1	28·5	89·4	5·04	33	371	509	0·2	11·1	15·4	92	98·4
Parkgate (S. Yorks.)	0·9	38·6	87·0	5·46	38	384	513	1·7	15·6	20·0	89	98·1
Silkstone (S. Yorks.)	1·3	35·7	86·8	5·47	38	376	495	1·4	16·7	14·9	—	—
F.3 (Scotland)	2·3	37·5	—	—	28	405	489	1·2	12·3	20·1	89	98·0
Barnsley (S. Yorks.)	2·8	34·5	85·1	5·24	28	401	481	1·4	12·9	20·4	79	96·9
A.4 (Scotland)	2·6	37·6	—	—	27	400	480	1·3	14·4	18·2	76*	96·4
Main (Northumberland)	4·7	41·0	85·1	5·48	26	409	480	1·1	16·7	20·3	75	97·3
Swallow Wood (S. Yorks.)	3·9	37·9	82·6	5·39	24	410	462	1·6	8·8	24·7	72	96·4
Waterloo (Derbyshire)	5·2	38·9	83·4	5·40	11	430 †	460 †	3·9 †	6·5†	25·4†	72*	95·6
E.3 (Scotland)	5·2	29·1	—	—	19	417	451	2·0	7·5	25·7	70	97·8
H.2 (Scotland)	5·6	38·5	84·1	5·64	21	397	482	2·3	13·3	18·4	65*	96·8

* Compressed charge. † Rate of heating 3° C. per minute.

to be the beginning of plasticity (*i.e.* when the resistance of the column of powdered coal to the passage of gas increased) was low, and the amount remaining in the coal when plasticity was assumed to have ceased (*i.e.* when the resistance of the coal to the passage of gas fell to a low value), decreased by from 1 to 8 per cent. when the rate of heating was increased from 1° to 5° C. per minute. Davidson's results for a rate of heating of 1° C. per minute are reproduced in Table LXXXIX, together with analyses of the coals.

These results show that there is no appreciable evolution of volatile matter before the temperature that indicates (according to Foxwell's method) the beginning of the plastic range. The volatile matter remaining at the end of the plastic range is low for the good coking coals and high for three of the poorest, but the amounts do not vary as regularly as might be expected for coals of intermediate quality owing, no doubt, to experimental error.

In later work (private communication) Davidson used a continuous method, similar to Audibert's, for determining the evolution of volatile matter, and his results for a rate of heating of 2° C. per minute are recorded in Table XC.

These results confirm the earlier in showing that the amount of volatile matter remaining at a temperature representing the end of the plastic range is least with the best and highest with the poorest coking coals, but the range of values for coals of intermediate quality is narrow, and the differences are insufficient to explain the marked differences in hardness of the cokes produced from the coals in practice. The experimental method, though an improvement on that used for the earlier work, is open to criticism on the grounds that but a small quantity of coal is used in the test and that some doubt attaches, as will be shown later, to the value of temperature taken as representing the end of the plastic range. Nevertheless, Davidson's method of test has proved of value in correlating the hardness (impact hardness) of cokes made from blends of coals in an experimental oven with their residual volatile matter at the end of the plastic range. (W. Davidson, *Gas World, Coking Section*, 1933, *Jan.*, p. 8; *Yearbook Coke Oven Man. Ass.*, 1934, p. 109).

B. Hofmeister (*Glcükauf*, 1932, **68**, 405), in similar work to Davidson's, has also used a continuous method of determining the volatile matter evolved from coal during heating, and a modification of Foxwell's method of measuring the plastic range. Whereas Davidson's modification retained Foxwell's horizontal tube with means of preventing expansion of the coal, Hofmeister, like most foreign users of Foxwell's principle, preferred a vertical tube in which the column of crushed coal was free to expand upwards. The temperature at which there was the maximum resistance to the passage of a stream of nitrogen was taken by Hofmeister to be the end of plasticity, the subsequent gradual decrease in resistance

TABLE XC.—THE LOSS OF VOLATILE MATTER FROM COAL DURING HEATING (DAVIDSON).

Coal	Moisture, per cent. air-dried	Vol. Matter, per cent. dry ash-free	Carbon (per cent. sulphur-dry, ash- and free)	Hydrogen	Caking Index	End of Plastic Range °C.	Volatile Matter and Plastic Range (see text)			Shatter Indices of Coke	
							Before	During	After	1½-in.	½-in.
Two Foot Nine (S. Wales)	0·8	24·0	89·9	4·66	33	540	0·8	9·9	9·9	97	99·0
D. (Scotland)	1·3	29·1	—	—	24	515	2·0	13·5	10·8	93	98·3
R 6 (Scotland)	1·2	30·6	—	—	26	507	1·9	12·3	12·8	90	97·7
R 1 (Scotland)	1·3	25·7	—	—	15	505	1·8	7·9	12·6	90	98·2
Silkstone (S. Yorks.)	1·0	36·0	86·8	5·48	44	502	2·0	16·0	12·0	86	97·3
Parkgate (S. Yorks.)	1·3	34·9	86·9	5·79	38	504	1·4	16·5	11·5	85	96·7
G. (Scotland)	1·3	29·2	—	—	24	512	1·4	12·2	11·3	83	98·0
Barnsley A. (S. Yorks.)	1·9	37·2	84·8	5·48	19	495	1·7	16·3	13·0	82	96·9
C 1 (Scotland)	2·1	34·0	—	—	23	502	1·3	14·5	13·4	78	97·0
L 2 (Scotland)	2·6	38·7	—	—	32	504	1·5	18·5	12·6	78	98·4
A (Scotland)	3·1	37·8	—	—	25	488	1·1	16·9	13·5	76	96·4
Barnsley C (S. Yorks.)	1·9	36·4	84·2	5·65	18	482	1·9	15·3	13·2	75	96·4
Barnsley B (S. Yorks.)	3·7	38·5	82·9	5·22	15	481	1·6	11·2	16·5	72	91·9
E (Scotland)	4·3	38·5	—	—	19	483	1·5	13·3	16·6	70	97·8
Thick (S. Staffs.)	5·4	37·6	81·4	5·63	1	430*	3·5	5·9	32·1	56	89·3

* Rate of heating 5° C. per min., not plastic when rate 2° C. per min.

as the temperature was raised being regarded as indicating a gradual extension of the zone of semi-coke from the wall of the tube towards the centre. Although this argument is unsatisfactory, in view of the fact that the temperature of the charge of coal should not differ by more than a few degrees between the walls of the tube and the centre, whereas the range of temperature over which there was a gradual fall in resistance to the passage of gas might be 80° C., it will be shown later that Hofmeister was justified in his choice of the temperature at which the resistance was at a maximum, under the conditions of his tests, as indicating the end of plasticity. Some of his results are recorded in Table XCI.

TABLE XCI.—THE PLASTICITY OF COAL AND THE EVOLUTION OF VOLATILE MATTER (HOFMEISTER).

Coal No.	Volatile Matter, per cent. ash-free	Caking Index	Plastic Range ° C.		Volatile Matter and Plastic Range (see text)		
			Beginning	End	Beginning	During	After
2	37·7	13	395	430	6·3	7·0	17·5
6	33·4	11	395	445	7·5	7·0	14·5
3	30·2	17	395	425	1·9	2·7	19·7
5	28·7	12	400	445	3·3	5·8	14·0
7	27·[illegible]	11	430	480	7·2	4·8	12·0
8	25·7	11	420	475	6·0	4·8	10·7
4	22·1	18	390	440	1·5	3·0	16·9

It will be noted that Hofmeister recorded greater amounts of volatile matter as being evolved before the plastic range was reached than did Davidson, but since he did not record the moisture-contents of his coals it is impossible to say how much of this "volatile matter" was moisture. With the narrower plastic range assumed by Hofmeister, the proportion of the volatile matter that would be evolved after plasticity had ceased was greater than in Davidson's tests. From graphs, Hofmeister showed that the rate of evolution of volatile matter was greatest just before and during the plastic range.

K. Bunte, H. Brückner and W. Ludewig (*Glückauf*, 1933, **69**, 765), in their study of the relationships between the plasticity of coal and the evolution of volatile matter, made simultaneous determinations of the plastic ranges (by Foxwell's method) and the yields of volatile matter (both gravimetric and volumetric). On calculating the rate of change of pressure with temperature during measurement of the plastic range, the maximum was taken to be at the temperature of maximum plasticity. This temperature was from 5° to 20° C. below the temperature of maximum resistance of the column of coal to the passage of gas (which was termed the

" semi-coke point "). The higher temperature at which the resistance fell to a low and uniform value was termed the " coke point " and was regarded as marking completion of the opening of closed pores. Some of the results of these investigators are recorded in Table XCII. The yields of volatile matter are gravimetric.

TABLE XCII.—THE PLASTICITY OF COAL AND THE EVOLUTION OF VOLATILE MATTER.
(BUNTE, BRÜCKNER AND LUDEWIG).

Kind of Coal	Moisture, per cent. air-dried	Vol. Matter, per cent. on ash-free dry basis	Plastic Range °C.		Semi-Coke Point °C	Coke Point °C	Evolution of Volatile Matter at:				Remained Vol. Matter
			Beginning (1)	Maximum (2)	(3)	(4)	(1)	(2)	(3)	4	
Gas (Saar)	1·3	33·9	382	391	407	460	4·4	1·2	2·7	8·0	15·7
Coking (U. Silesia)	1·5	30·2	385	402	407	490	5·2	1·2	6·0	10·8	10·3
Coking (Wurm)	1·0	19·9	410	445	464	520	3·9	1·6	1·0	3·3	7·5

The evolution of gas at temperature (1) was only 8·0, 4·5 and 5·5 c.c. per gramme for the three coals.

Between the temperature taken as marking the beginning of plasticity and the " semi-coke point " the evolution of volatile matter was small and bore no relationship to the total volatile matter content of the coal. Between the " semi-coke point " and the " coke point " the evolution of volatile matter was considerable for the two coals of higher volatile matter content.

In general, the work of different experimenters shows that the decomposition-point of a coal as indicated by the evolution of gas is almost identical with its softening-point, but that the evolution of liquor and tar may occur at a lower temperature. It is difficult to reconcile the results of different experimenters regarding the evolution of volatile matter within the plastic range, because different interpretations are placed on the results of tests, based on Foxwell's method, designed to locate the plastic range.

The Evolution of Volatile Matter from Coal at Constant Volume.—Before considering methods of studying the plasticity of coal further, it is desirable to discuss the results of tests in which the close relationship that exists between plasticity and the evolution of volatile matter has been established.

When a charge of coal is coked in a coke-oven, the coke is formed under conditions of almost constant volume, the tendency for a layer of coal at any particular position in the charge to swell being neutralised by the same tendency of the coal above and below it so that there is but little difference between the volumes of the original

charge of coal and of the resultant coke. The air-space between the particles of coal in an oven charge amounts to about one-third of its apparent volume. This air-space may be filled as the coal expands during heating, but the ability of some coals to swell to twice or three times their original volume is restrained.

Tests have been made on the evolution of gas from coal during heating when the expansion of the coal was prevented by enclosing it in a retort fitted with a steel plunger fixed in position by a screw-on cap (see Fig. 83). The retort was of steel $\frac{1}{8}$-in. in diameter and the plunger had a clearance of 1/64-in. In the tests, $2\frac{1}{2}$-gramme

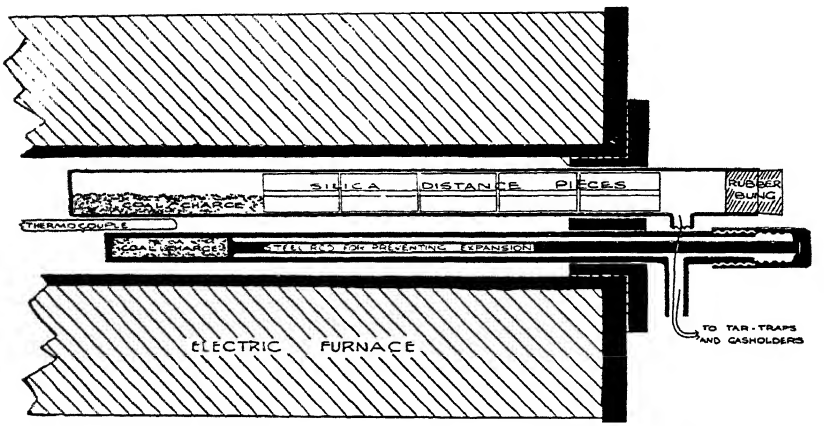


FIG. 83.—Apparatus for Studying the Evolution of Gas from Coal (*a*) Free to Swell, (*b*) Prevented from Swelling.

charges of coarsely-ground coal, through $\frac{1}{8}$-in. mesh, were raised in temperature at a rate of 1° C. per minute. Parallel tests were made with charges of 10 grammes of coal in a silica retort (Gray-King apparatus) heated in the same furnace. Both retorts were connected to trains of condensers and gas-holders and the rates of evolution of gas were determined.

It was found that, whilst gas was freely evolved above about 350° C. from coal free to expand, when that coal was prevented from expanding hardly any gas was collected below about 500° C. It was then evolved suddenly in volume equal (per gramme of coal) to that gradually collected up to 500° C. from the charge that was free to expand. A number of records of tests are given in Fig. 84 (see R. G. Davies and R. A. Mott, *Fuel in Science and Practice*, 1933, **12**, 378), the evolution of gas being expressed per 10 grammes of coal. The temperature at which there was a sudden evolution of gas from a charge at constant volume was, in general, higher the higher the rank of the coal. The sudden evolution of gas occurred over a temperature-range of 10° C. and appeared to be due to the imprisonment

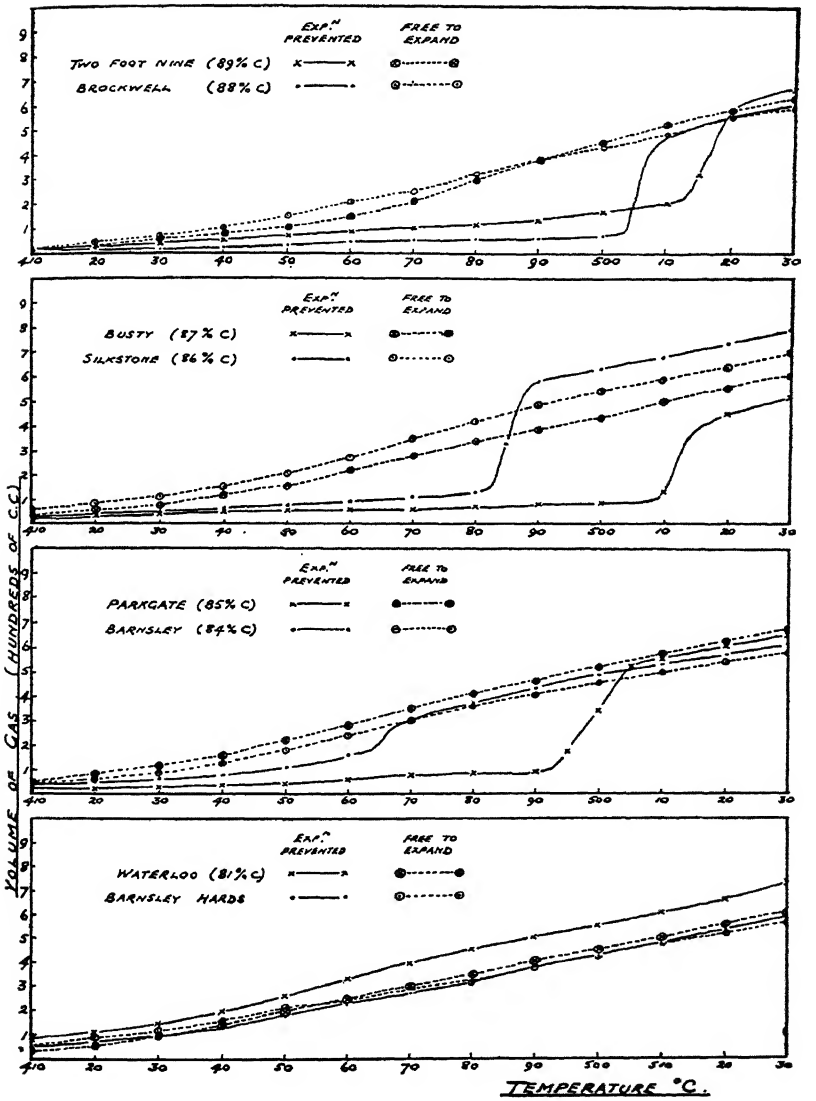


FIG 84.—Evolution of Gas from Coals (*a*) Free to Swell, (*b*) Prevented from Swelling.

of gas whilst the coal was plastic and its release as soon as plasticity was destroyed. For non-swelling coals and for Barnsley hards the curves for the evolution of gas were similar whether the charge was or was not free to expand.

In later work, the evolution of liquor and tar from a charge of coal heated at constant volume was studied. A series of tests

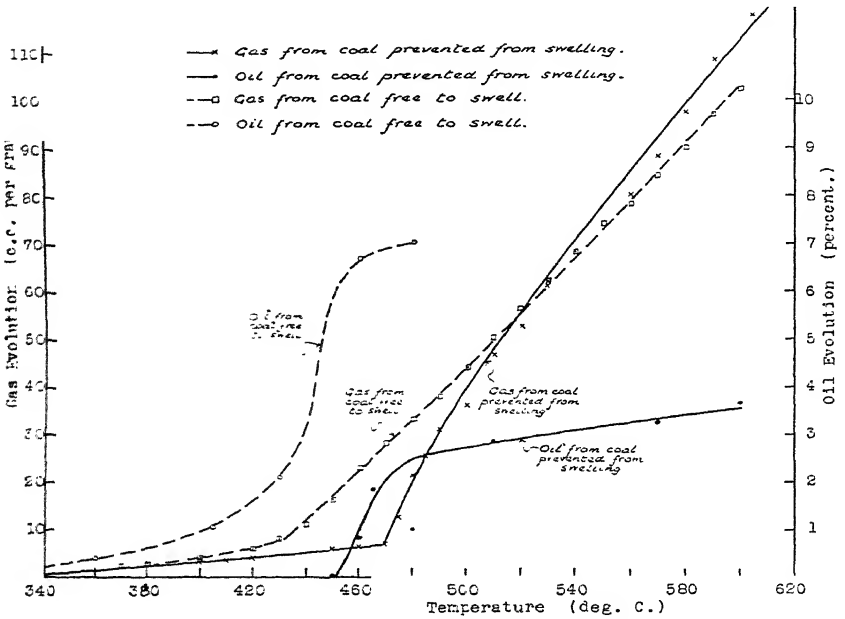


FIG. 85.—Evolution of Gas and Tar from Coal (*a*) Free to Swell, (*b*) Prevented from Swelling.

was made with 5-gramme charges of coal heated to different temperatures below and above that of sudden evolution of gas, as previously determined. The results are recorded in Fig. 85. The coal had been partly oxidised by exposure to air, and the sudden release of gas occurred at a lower temperature than for the fresh coal, but it is clear that the evolution of tar was delayed until the release of gas. In other tests, in which larger charges of coal were used (see Fig. 86), the total volatile matter remaining in Parkgate coal heated at constant volume at 1° C. per minute to different temperatures was found to be:

Temp. ° C.	350	400	450	500	550
Vol. Matter remaining	34·1	30·5	29·8	19·6	18·8

The temperature at which a sudden evolution of gas occurred was 495° C.

For Brockwell coal the amount of gas suddenly released during heating at constant volume amounted to 100 c.c. from $2\frac{1}{2}$ grammes. Assuming free air-space of 33 per cent. (0·8 c.c.), the pressure of

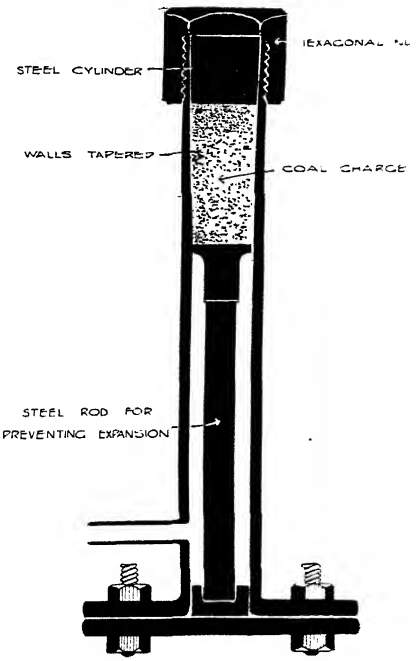


FIG. 86.—Apparatus for Studying the Quality of Coke made from Coal Prevented from Swelling.

gas within the charge just before its release would be about 125 atms., or 1,800 lb. per sq. in., and, if the vapour-pressure of liquor and tar be included, the total pressure which plastic coal can retain might be as high as 5,000 lb. per sq. in. Until the pressure is released the free evolution of volatile matter is prevented. It will be shown later that the temperature at which the plasticity of coal ceases is about 80° C. lower when the coal is free to expand than when expansion is prevented. When determining the relationship between the "plastic range" of a coal and the evolution of volatile matter in separate series of tests it is therefore important that the conditions of heating should be the same. In both Foxwell's and Davidson's work the plastic range was determined with the charge of coal prevented from expanding, and the evolution of

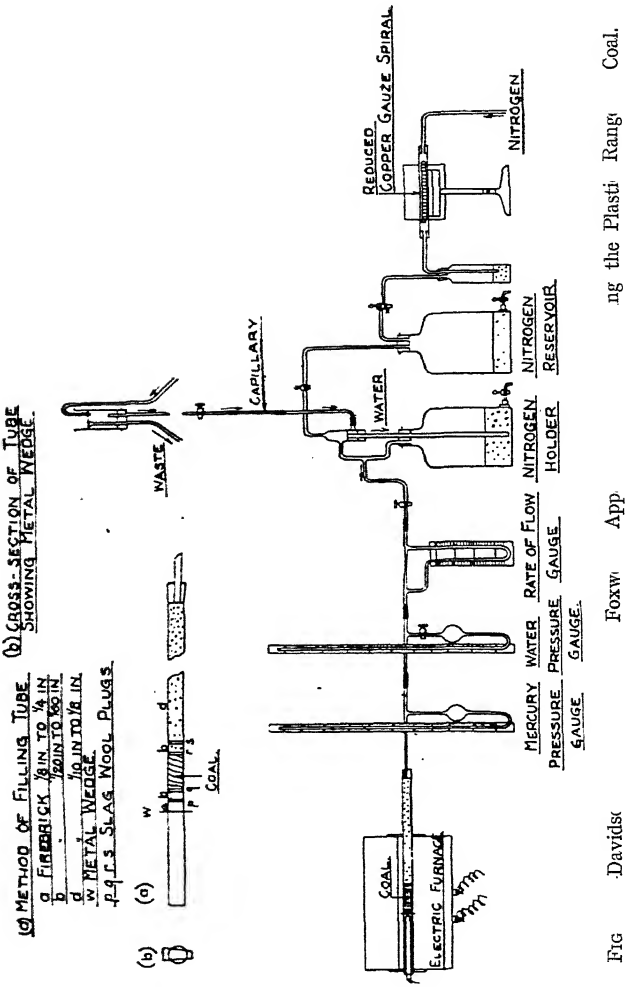


Fig. 87. Davidson's Apparatus for Determining the Plastic Range of Coal.

volatile matter with the coal free to expand, but in the work of Bunte, Brückner and Ludewig the coal was free to expand in both series of tests.

The fact that plastic coal could be quite impermeable to the passage of gas was emphasized by Davidson (*Fuel in Science and Practice*, 1930, **9**, 489), who, in his modification of Foxwell's method of determining the plastic range (Fig. 87), introduced a flow-gauge and noted the temperature at which a negative pressure, indicating

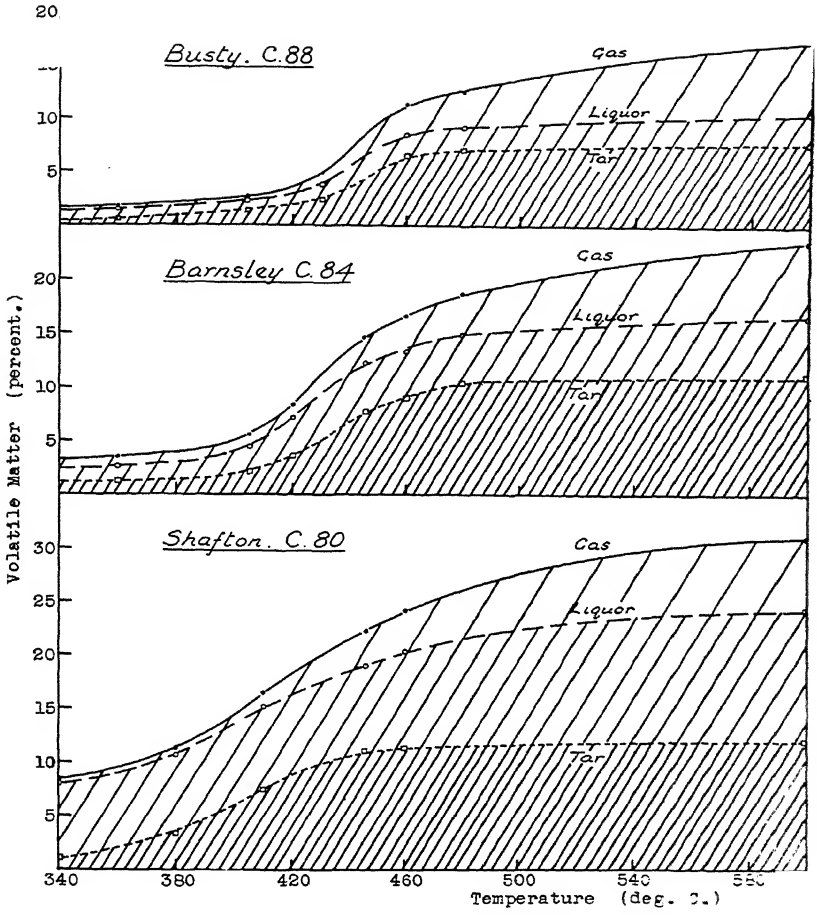


FIG. 88.—The Quality of the Volatile Matter from Coals of Different Carbon Content, and at Different Temperatures.

a reversal of the flow of nitrogen, occurred. This temperature he took to be that at which plasticity of the coal began.—R. G. Davies and R. A. Mott (*Fuel in Science and Practice*, 1933, **12**, 380), in determinations of the plastic range for a number of coals by Foxwell's method, forced a stream of nitrogen through the column of coal under a head of 14 ft. (about ½-atm.). Since the pressure of gas within a charge of plastic coal may be several atmospheres, the complete stoppage of the stream of nitrogen when

the coal was plastic is not surprising. The results obtained by Davies and Mott are recorded in Table XCIII, the rate of heating being 2° C. per minute and the charge being kept at constant volume.

TABLE XCIII.—THE PLASTIC RANGE OF COAL (FOXWELL'S METHOD).

Coal	Carbon, per cent. Parr's basis	Sheffield Lab. Coking Test		Foxwell's Test			
		Softening Temp. ° C.	Expansion Temp. ° C.	Temp. of Initial Resistance ° C.	Temp. of Maximum Resistance ° C.	Temp. of Final Resistance ° C.	Maximum Resistance, mm. W.G.
Two Foot Nine (S. Wales)	89·7	383	429	425	510	580	4,800
Busty (Durham)	88·7	362	407	405	500	590	4,560
No. 5 (Kent)	87·5	363	412	405	500	580	4,670
Parkgate (S. Yorks.)	86·9	342	395	405	495	550	4,280
Halifax Soft (S. Yorks.)	86·8	346	395	395	490	590	4,620
Mountain Mine (Lancs.)	86·6	354	414	410	490	540	4,560
Silkstone (S. Yorks.)	85·5	347	400	385	490	540	4,500
Arley (Lancs.)	85·4	—	—	400	485	550	4,080
Barnsley (S. Yorks.)	85·1	394	406	410	460	480	1,020
Black Shale (Derby.)	84·2	343	388	390	475	540	4,020
Deep Hard (Derby.)	83·1	361	—	420	460	480	80
Waterloo (Derby.)	83·0	320	—	420	450	480	140
Swallow Wood (S. Yorks.)	82·3	351	—	410	450	480	180

Since with the strongly swelling coals, containing over 86 per cent. carbon, the plastic layer was impermeable, the measured maximum resistance to the passage of a stream of nitrogen is of little significance, being merely that of the head of water available for the test. The slope of a curve relating the pressure of the nitrogen to the temperature of the coal indicates the rate at which the pressure rises as the coal becomes plastic and varies with the capacity of the apparatus, as does the rate at which the pressure falls from the maximum as plasticity is destroyed with rise of temperature. In the tests recorded in Table XCIII, it was possible to pass nitrogen through the cold column of coal at a rate of 20 c.c. per minute, but as soon as the coal became plastic and impermeable to the stream of nitrogen the rate of increase of pressure

within the apparatus was dependent upon the release of gases from the plastic coal.

For example, Foxwell reported his results by graphing values of temperatures against the relationship $m = \frac{ps}{vl}$, where p is the water-gauge pressure in mm. required to pass v c.c. of nitrogen per minute through a column of coal of cross-sectional area s sq. cm. and length l cm. With apparatus having a capacity of 645 c.c., the water-gauge reading for the Parkgate coal at 460° C. was 2,480 mm. The column of coal was 2·5 cm. long and 0·75 sq. cm. in cross-sectional area. A tangent to the pressure-temperature curve for the test showed that at 460° C. the rate of increase of pressure was 82 mm. per minute. Under the total pressure represented by the barometric, the water-gauge and the instantaneous value of the rate of increase, the volume of nitrogen in the apparatus would be reduced from 645 to 636 c.c., so that a flow of 9 c.c. should be required to maintain the recorded increase of pressure. The measured volume of water used was 9·3 c.c., so that 0·3 c.c. (0·28 c.c. at N.T.P.) of nitrogen was passing through the column of coal. Tests of the rate of evolution of gas (including steam) from the Parkgate coal when raised in temperature (free to expand) at a rate of 2° C. per minute showed that at 460° C. the rate was 7·2 c.c. per minute. Assuming (with Foxwell) that half of this volume of gas passes through the whole of the coal, the total volume of gases passing at 460° C. during the determination of plasticity was 3·6+0·28 c.c. per minute and $m = 434·6$. The volume of gas released from, and assumed to be passing through, the plastic coal was 93 per cent. of the total, so that the calculation of m is dependent essentially on the amount of gas evolved from the coal.

It has been shown that gas is released suddenly from plastic coal that is prevented from swelling at a temperature which is presumably the end of the "plastic range." Such a sudden release of gas should determine the attainment of a maximum pressure during Foxwell's test. That this is so is shown by the following comparison :

Coal	Carbon content, per cent. dry, ash-free basis	Coal Heated at Constant Volume	
		Temp. of Rapid Evolution of Gas, ° C. (Davies & Mott)	Temp. of Max. Resistance to Passage of Gas, ° C. (Foxwell's Test)
Two Foot Nine (S. Wales) .	89	515	510
Busty (Durham) . . .	88	510	500
Parkgate (S. Yorks.) . .	85	495	495
Silkstone (S. Yorks.) . .	86	485	490
Barnsley (S. Yorks.) . .	84	465	460

If, during Foxwell's test, the temperature at which the resistance to the passage of nitrogen is at a maximum is regarded as the end of the plastic range, the significance of the subsequent fall in resistance requires consideration.

The downward slope of the resistance curve from the maximum is usually gradual at first (owing, no doubt, to the continued evolution of gas from the coal), but then becomes steep until it falls to a low value, when it again becomes gradual. Over the steep portion of the curve the rate of fall of pressure varies from 142, 170 and 180 mm. per minute, for the Halifax Soft, Mountain Mine and Busty coals, to 270 and 360 mm. per minute for the Black Shale and the Parkgate. For such a fall in pressure to occur with the Parkgate coal, the volume of gas within the apparatus would have to decrease at a rate of 19 c.c. per minute, and since the capillary controlling the rate of flow of gas through the apparatus did not admit of the passage of more than 20 c.c. of nitrogen per minute, the fall of pressure observed was about the maximum possible.

The final gradual fall in pressure appears to be influenced by the final evolution of tar from the coal, which may clog the cellular structure of the coke or the interstices of the crushed fire-brick used to hold the charge in position. Of all the significant temperature-measurements, that at which the pressure falls to a low value is most influenced by the rate of heating, as the following records show:

	Two-Foot Nine			No. 5, Kent			Parkgate		
Rate of Heating, ° C. per min.	1	2	4	1	2	4	1	2	4
Temp. of initial increase of resistance ° C.	425	425	435	410	400	415	415	415	420
Temp. of maximum resistance ° C.	485	510	550	480	500	540	465	495	520
Temp. of final fall to low resistance ° C.	550	580	700	540	580	720	480	550	650

The markedly high temperature at which the resistance of the column of coal falls to a low value when the rate of heating is 4° C. per minute cannot reasonably be ascribed to a critical stage in the formation of coke, but is merely a feature of the apparatus and experimental method.

Before leaving consideration of Foxwell's method of studying the plastic range, attention may be drawn to the relationship between the temperature at which the resistance of a column of coal to the passage of a stream of nitrogen increases, and the temperature of initial expansion of that coal as determined in the Sheffield Laboratory Coking Test (see Table XCIII).

The Quality of the "Volatile Matter" Evolved from Coal at Different Temperatures

The total amount of volatile matter evolved from a coal has been shown to be influenced by the conditions of carbonisation, the rate and duration of heating and the freedom given to the coal to swell being the most important variables. Liquor and tar (oils) may be evolved from a coal before there is active decomposition, of which the best indication is the rate of evolution of gases. The evolution of gases at a rate exceeding 5 c.c. per gramme of coal may be established by prolonged heating at temperatures *circa* 350° C., but may be delayed until a temperature of 500° C. is attained if the rate of heating exceeds 1° C. per minute and the coal is prevented from swelling. The distillation of tar from the charge in any quantity is dependent on the amount of gas evolved exceeding about 5 c.c. per gramme.

The quality of the volatile matter evolved from a coal depends upon its rank, as is shown in Fig. 88 for three coals of 88, 84, and 80 per cent. carbon-content heated at a rate of 1° C. per minute in the Gray-King assay apparatus (see Allinson and Mott, *loc. cit.*). At the lowest temperatures, liquor is the principal constituent of the volatile matter, with some oil but with little or no gas. At a higher temperature, the principal constituent of the volatile matter is tar, but its evolution ceases at about 470° C., and the production of liquor above that temperature is low. The total amount of volatile matter evolved at 360° C. is as high as 10 per cent. for the Shafton coal (80 per cent. carbon), as compared with 3·5 and 1·8 per cent. from the coals of higher rank, but 80 per cent. of the volatile matter from the Shafton coal is water.

The liquor evolved from a coal may occupy a large volume as steam and may be as effective as gas in removing oils mechanically. A considerable amount of liquor is evolved from a coal between its temperature of softening and swelling, as indicated in Fig. 88, whilst below the temperature of softening much liquor may be evolved (see Table XCIV).

These records show that from coals of low rank a considerable amount of liquor is evolved below the temperature of softening, but that the evolution of tar and gas is usually negligible. With the coals that swell, but little liquor is evolved between the temperature of softening and of swelling, but there is always some tar and the evolution of gas is evident. During swelling, there is a tendency for the amount of gas evolved to increase.

Since the evolution of gas and vapour must be a primary cause of the swelling of a coal, it is of interest to calculate their volumes over different ranges of temperature. This is done for three coals in Table XCV, liquor being calculated as steam, and tar vapours being assumed to have a molecular weight of 128.

TABLE XCIV.—THE QUALITY OF THE VOLATILE MATTER EVOLVED FROM COAL.

Coal	Below Temp. of Softening			Between Temp. of Softening and Swelling			During Swelling *		
	Liquor, per cent.	Tar, per cent.	Gas, cc. per cent.	Liquor, per cent.	Tar, per cent.	Gas, cc. per cent.	Liquor, per cent.	Tar, per cent.	Gas, cc. per cent.
Brockwell (89% C.) †	1·1	0·3	nil	nil	1·3	1·6	0·3	1·0	7·0
Busty (88% C.)	1·4	0·4	nil	0·1	0·7	2·9	0·1	1·0	11·7
Silkstone (86% C.)	2·1	nil	nil	nil	1·2	1·0	nil	1·9	7·9
Parkgate III (85% C.)	1·1	nil	nil	nil	1·9	4·7	0·2	0·8	2·0
Parkgate II (84% C.)	2·2	0·5	0·4	0·3	1·4	3·3	0·2	0·6	2·4
Barnsley IV (84% C.)	1·5	1·2	0·9	1·3	0·8	2·2	0·5	1·7	4·9
Fenton (83% C.)	3·8	nil	nil	0·1	4·1	4·3	0·1	1·7	8·1
Swallow Wood (82% C.)	4·0	0·2	1·0	(Do not swell)					
Waterloo (81% C.)	5·1	nil	nil						
Shafton (80% C.)	8·1	nil	nil						
Thick (79% C.)	14·0	nil	nil						
Main (77% C.)	11·4	nil	4·4						

* In the Sheffield Laboratory Coking Test.
† Carbon expressed on dry, ash-free basis.

For the two coking coals, Busty and Barnsley, the most marked evolution of volatile matter occurs over the range 420–480° C., *i.e.* just after the swelling-range. At 480° C., most of the liquor and tar has been evolved, but the rate of evolution of gas has increased and this increase is maintained as the temperature is raised. It is noteworthy that over the swelling-range, 360–420° C., the volume of gas and vapour is greater the lower the rank of the coal. The volume of gas and vapour evolved is therefore no criterion of the swelling-power of a coal though, as will be discussed in a later chapter, the evolution of gas is essential for swelling.

It should be noted here that the conditions of heating in the Gray-King assay, used for measuring the evolution of volatile matter at different temperatures, and in the Sheffield Laboratory Coking Test, used for measuring the softening and swelling ranges, are not strictly comparable even though the coal is free to expand in both, and the rate of heating may be the same. In the Gray-King assay, the charge of coal is horizontal, and a space is left above

it for the release of volatile matter, so that it is free to expand upwards. There may therefore be lateral swelling at a higher temperature than in the Sheffield Laboratory Coking Test, in which the charge of coal is vertical. The results of parallel experiments illustrating this are recorded in Table XCVI.

TABLE XCV.—THE EVOLUTION OF VOLATILE MATTER FROM COAL DURING DIFFERENT RANGES OF TEMPERATURE.

Temperature-Range, ° C.	Below 360	360–420	420–480	480–600	Total to 600
BUSTY					
Total volatile matter, per cent.	1·8	2·2	8·5	5·1	17·6
Total gas and vapour, c.c. per gramme	17·5	13·0	40·2	68·5	139·0
Gas, c.c. per gramme	nil	9·0	29·0	52·6	90·6
BARNSLEY					
Total volatile matter, per cent.	3·5	4·6	10·8	4·5	23·4
Total gas and vapour, c.c. per gramme	21·0	21·0	63·3	52·2	157·5
Gas, c.c. per gramme	1·0	9·2	23·5	40·5	72·2
SHAFTON					
Total volatile matter, per cent.	10·0	8·2	7·8	5·2	31·2
Total gas and vapour, c.c. per gramme	103·0	28·8	46·4	73·3	251·5
Gas, c.c. per gramme	2·0	17·0	22·0	36·9	77·9

TABLE XCVI.—A COMPARISON OF THE SWELLING-RANGES OF VERTICAL AND HORIZONTAL CHARGES OF COAL.

Coal	Temp. of Initial Swelling, ° C.		Temp. of Final Swelling, ° C.		Swelling, per cent.	
	Vertical	Horizontal	Vertical	Horizontal	Vertical	Horizontal
Silkstone	407	414	428	433	80	112
Parkgate	393	400	413	418	120	180
Black Shale	384	391	403	417	168	362
Tupton	399	408	416	417	93	57

The swelling is recorded as beginning at a temperature 7 to 10° C. lower when the charge is vertical than when it is horizontal, and as ending at a temperature up to 14° C. lower. The differences

are, however, not sufficient to invalidate the conclusion drawn from Table XCV.

The Rate of Production of Gas from Different Coals

Although the coals of lowest rank begin to evolve gas at the lowest temperatures, the greatest volumes of gas at higher temperatures are yielded by coals of high rank. After the de-

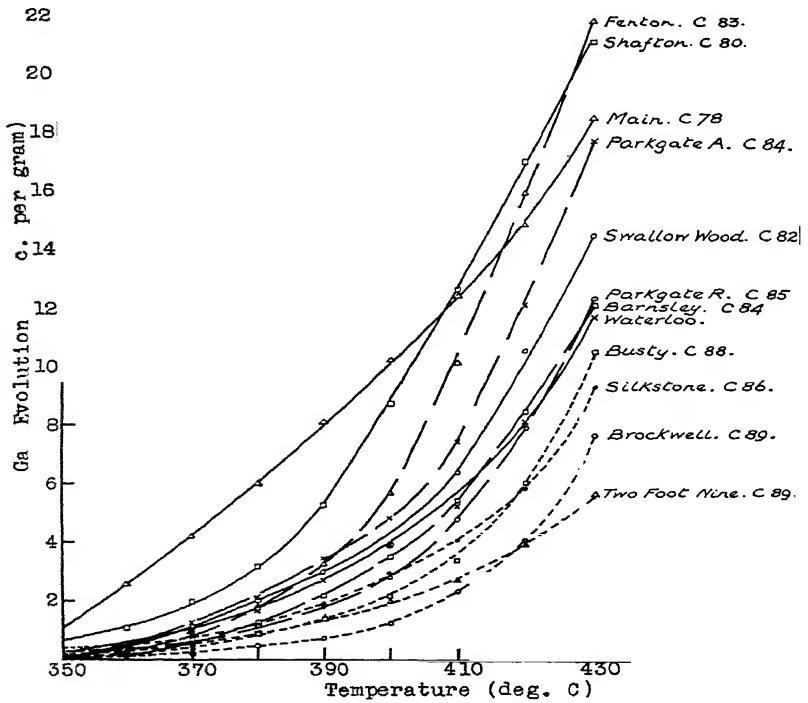


Fig. 89.—Total yields of Gas from Coals of Different Carbon Content over the Range 350–430° C. at a Rate of Heating of 1° C. per min.

composition-point has been passed, the production of gas from a coal raised in temperature at a rate of 1° C. per minute is small until the temperature exceeds about 420° C. This is illustrated in Table XCVII, which records the volumes of gas evolved per gramme of coal over ranges of 10° C. when heated in the Gray-King assay apparatus at a rate of 1° C. per minute.

The temperatures at which a regular increase in the rate of production of gas occurred were 350–355° C. for the first four coals in Table XCVII, 335–355°C. for the next six and 320–325° C.

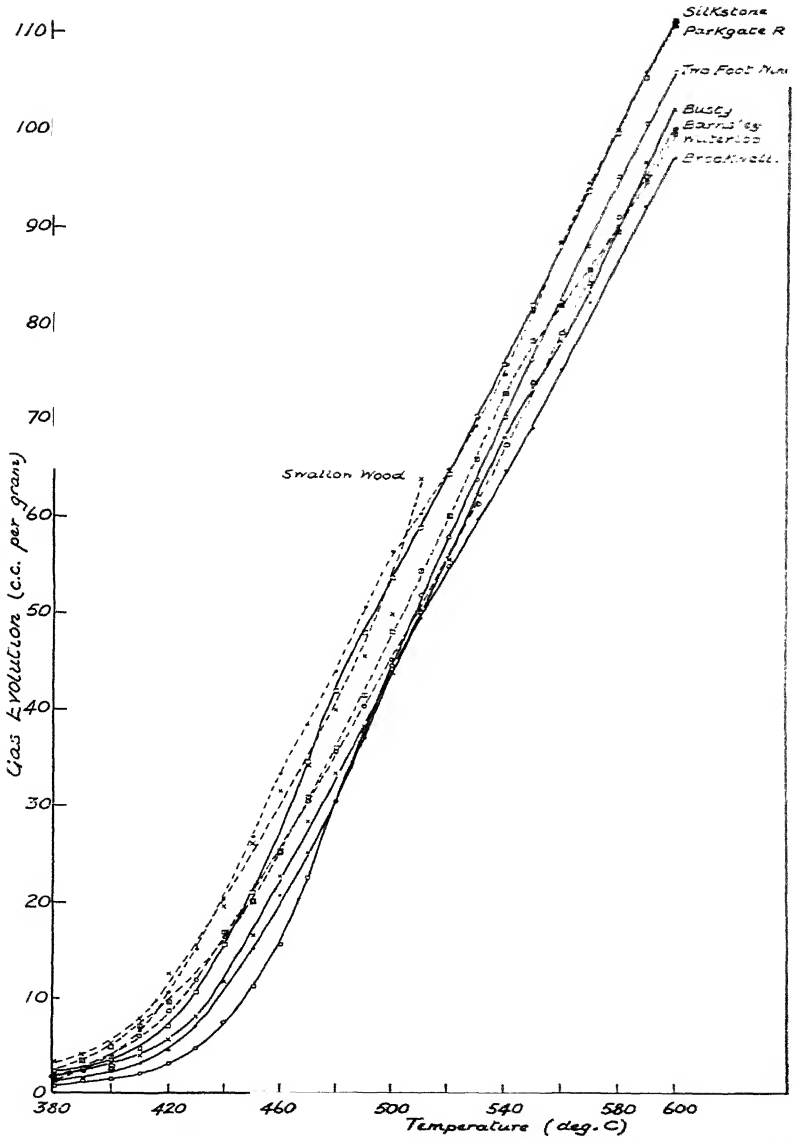


FIG. 90.—Total Yields of Gas from Coals of Different Carbon Content over the Range 380–600° C. at a Rate of Heating of 1° C. per min.

for the remaining two. The rates of production of gas, which were slow until a temperature of 390–410° C. had been passed, are indicated in Fig. 89. From the values for the production of gas within the swelling-ranges, it would appear that swelling, in the Sheffield Laboratory Coking Test, begins when the rate is about 1 c.c. per gramme per 10° C. rise in temperature, and ends when the rate is rapidly increasing to about 3 c.c. per gramme.

TABLE XCVII.—THE PRODUCTION OF GAS FROM COAL HEATED AT A RATE OF 1° C. PER MINUTE. C.C. PER GRAMME.

Coal	Upper Limit of Temperature-Range (° C.)											
	350	360	370	380	390	400	410	420	430	440	450	460
Two Foot Nine (89% C	0·1	0·1	0·3	0·4	0·5	·6	0·7	1·2	(1·7)	(3·0)	(3·6)	4·7
Brockwell (88% C.)	nil	nil	0·2	0·3	0·2	·6)	(1·0)	(1·8)	3·5	5·3	5·6	6·3
Busty (88% C.)	nil	0·1	0·3	0·5	0·5	·8)	(1·2)	(2·6)	(4·4)	5·3	5·8	5·9
Silkstone (86% C.)	0·1	0·3	0·4	0·4	0·6	·1)	(1·2)	(1·7)	3·4	5·3	5·9	6·7
Parkgate R (85% C.)	0·1	0·3	0·4	0·4	0·7	·9)	(2·0)	3·1	4·3	5·3	5·5	5·4
Parkgate A (84% C.)	0·2	0·4	0·7	0·8	1·3	·4	(2·6)	4·6	5·5	6·2	5·7	6·3
Barnsley (84% C.)	nil	0·2	0·5	0·6	0·9	·3)	(1·9)	(3·0)	3·6	3·2	3·6	4·1
Fenton (83% C.)	0·1	0·3	0·6	0·8	(1·5)	·4)	(4·4)	5·7	5·8	3·9	3·3	3·3
Swallow Wood (82% C	0·2	0·3	0·6	0·9	1·0	·9	2·5	4·1	3·8	3·7	4·7	4·5
Waterloo (81% C.)	0·2	0·3	0·5	0·7	1·0	·2	1·3	2·9	3·5	3·6	3·8	4·6
Shafton (80% C.)	0·5	0·6	0·9	1·2	2·1	·4	3·8	4·3	4·1	3·8	4·0	3·7
Main (77% C.)	1·3	1·3	1·0	1·0	2·0		1	2·4	3·6	3·6	4·8	4·4

Note.—The values in brackets lie within the temperature-range for swelling as determined in the Sheffield Laboratory Coking Test.

The total amounts of gas evolved over the range 420–600° C. for a number of coals heated at a rate of 1° C. per minute are recorded in Fig. 90. The decomposition in each instance is seen to be regular, and the total amount of gas produced up to 600° C. is about the same for all the coals although their volatile matter contents range between 22 and 37 per cent.

The total amount of gas produced up to 900° C. is shown in Fig. 91 for the Kent Thick coal heated at rates of 1, 1·6 and 5° C. per minute. It will be seen that 60 per cent. of the gas was evolved above 600° C., whilst at 420° C., when for most coals swelling has ceased, less than 5 per cent. had been evolved.

Some figures are available for one coal, Parkgate R, for the total amounts of gas and of steam evolved over successive ranges of temperature of 10° C., the coal being heated at a rate of 2° C. per minute, as follows:

Upper Limit of Temperature-Range ° C.	370	380	390	400	410	420	430	440	450	460	470	480	490
Gas, c.c. per gramme	—	—	0·5	0·3	0·8	0·8	2·1	3·1	5·3	5·5	7·5	6·9	6·5
Steam, c.c. per gramme	1·0	1·0	4·0	5·0	5·0	3·0	5·0	4·0	5·0	6·0	7·0	6·0	5·0

The volume of steam evolved at low temperatures is several times the volume of gas, but at higher temperatures the volumes are about equal. With coals of low rank, however, the amount of steam evolved is greater throughout, and it may exceed the amount of gas even at high temperatures. The effect of this steam on the early removal of adsorbed oils during the distillation

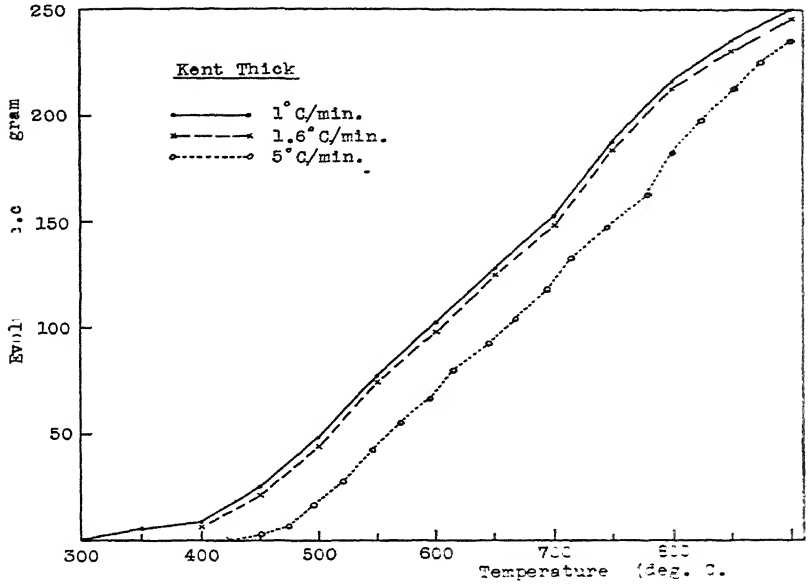


FIG. 91.—Total Yields of Gas to 900° C. at Different Rates of Heating of Kent Thick Coal.

of coals of low rank (*see* Figs. 78 and 79) may be considerable, and may account for their poor coking properties. This subject will be discussed further in a later chapter, when the effect of oil on the formation of coke is considered in detail.

CONCLUSIONS

The principal conclusions to be drawn from the work described in this chapter, as regards the relationship between the plasticity of coal when it is heated and the evolution of volatile matter, are :

(1) The volatile matter first evolved from coal when it is gradually raised in temperature consists mainly of liquor and tar, the amount of liquor increasing with decrease in rank of the coal. With a rate of rise of temperature of 1° C. per minute, but little tar (oil) is distilled until extensive decomposition of the coal substance (as indicated by the evolution of gas) has begun. Oils are, however,

liberated at a lower temperature and are adsorbed on the surfaces of the particles of coal, whence they are removed by entrainment when the rate of evolution of gas becomes sufficiently rapid.

(2) The rate of production of gas from different coals over the range of temperatures used for coke-making is about the same, but oil is distilled less readily from coals of higher rank because its adsorption is more extensive and persists to a higher temperature.

(3) The softening of coal does not precede the evolution of gas, and the coal begins to swell when but little volatile matter has been evolved. With coals of low rank, however, the evolution of liquor below the softening temperature may be considerable.

(4) In the Sheffield Laboratory Coking Test a coal swells over a narrow temperature-range, but may remain plastic up to 25° C. above the temperature at which swelling ceases. The principal evolution of tar occurs towards the end of the swelling-range, or just after swelling has ceased, the removal of tar being, no doubt, a factor in the loss of plasticity.

(5) If the swelling of a coal is restrained, loss of volatile matter is also restricted and plasticity persists to a higher temperature. A sudden release of gas then marks the end of plasticity.

328° C.

363° C. 31.

333° C. 2.

373° C. 56.

338° C. 6.

383° C. 80.

343° C. (Softening).

393° C. (Initial Expansion) 92.

353° C.

403° C. 100.

FIG. 93.—Appearance of Residues from Columns of Particles Heated to Various Temperatures in the Sheffield Laboratory Coking Test. R.M. Parkgate Coal. (The Coalescence Index is the percentage remaining on 60-mesh after dropping three times through a height of 2 ft.)

CHAPTER XV

THE SOFTENING AND SWELLING OF COAL

In the preceding chapter, the decomposition of coal as revealed by the evolution of gas, tar and liquor has been discussed with incidental reference to the phenomena of softening and swelling which accompany the decomposition. The softening and swelling which occur when coal is heated, either as a single lump or as a collection of particles, charged loosely into a retort or briquetted, can now be considered more closely.

The Softening-Point of Coal

The "softening-point" of a coal can be gauged by the temperature at which a column of particles, either loosely charged into a vertical tube or compressed as a pellet, contracts when subjected to a light load. In the work now to be described, the softening-points were determined under the conditions of the Sheffield Laboratory Coking Test, in which a charge of 6 grammes of coal of through 60-mesh size was raised in temperature at a rate of 1° C. per minute in a silica retort, 1·6 c.m. in diameter, under a load of 100 grammes (see Fig. 92).

The significance of the softening-point can be judged from the photographs in Fig. 93, which show the appearance of charges of R.M. Parkgate coal that had been heated below and above its softening-point (343° C.). Relative "coalescence indices" (obtained by a small-scale test) for the charges are recorded beneath each photograph, and show that coalescence of the particles of coal did not occur to any marked extent until they had been heated between 373° and 393° C., *i.e.* 30° and 50° C. above the softening-point. From the appearance of the caked products also, it is evident that the individual particles had not lost their identity at the softening-point.

The coalescence of the particles into aggregates is due to the distension of each individual particle that occurs above about 340° C. (when the temperature is raised at a rate of 1° C. per minute). This distension cannot readily be detected in small particles, but becomes obvious when lumps of the coal are heated. Figs. 94 and 95 show 1-inch cubes of R.M. Parkgate coal that have been heated to and above the softening-point. Each cube was measured and a cut face was ground on it for treatment with plaster of paris

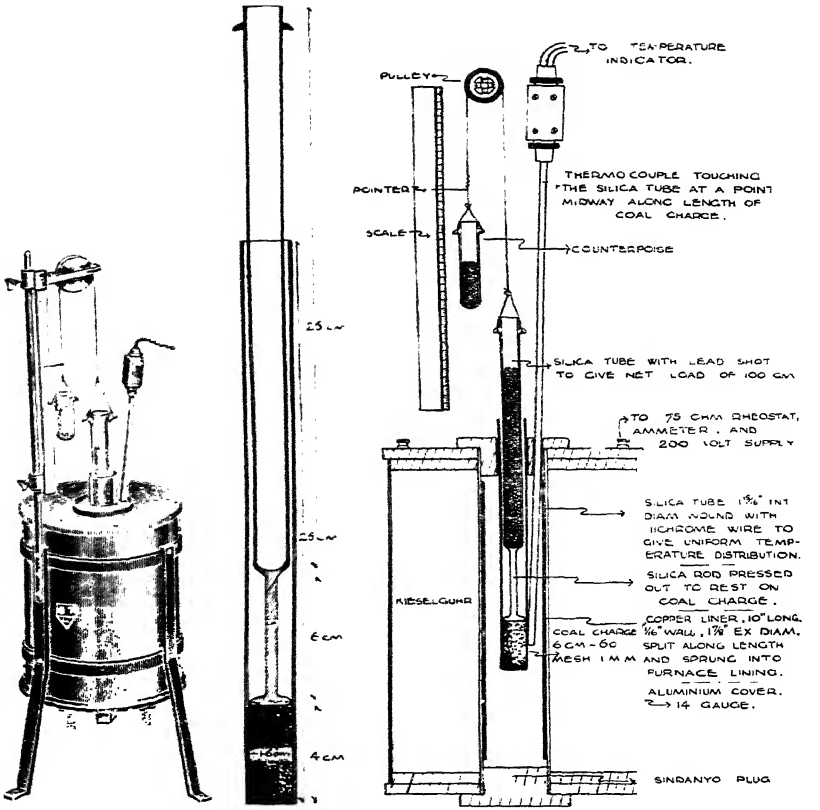


FIG. 92.—Silica Apparatus for Sheffield Laboratory Coking Test.

to disclose the structure. It will be seen that near the softening-point (343° C.) there was incipient formation of pores and that their formation was complete at 368° C. Distinction must be drawn between a true pore, which is approximately spherical in shape and has a well-defined boundary, and a cavity which may be a crack along the bedding-plane of the coal and has an ill-defined boundary. It may be noted that the pores, at first, had their longer axes parallel with the bedding-plane. At higher temperatures, however, the pores were so distended that their relation to the bedding-plane could not be discerned, whilst at still higher temperatures they began to disrupt.

The swelling-power of pieces of coal (R.M. Parkgate brights) of different sizes was determined by arranging them on a silica

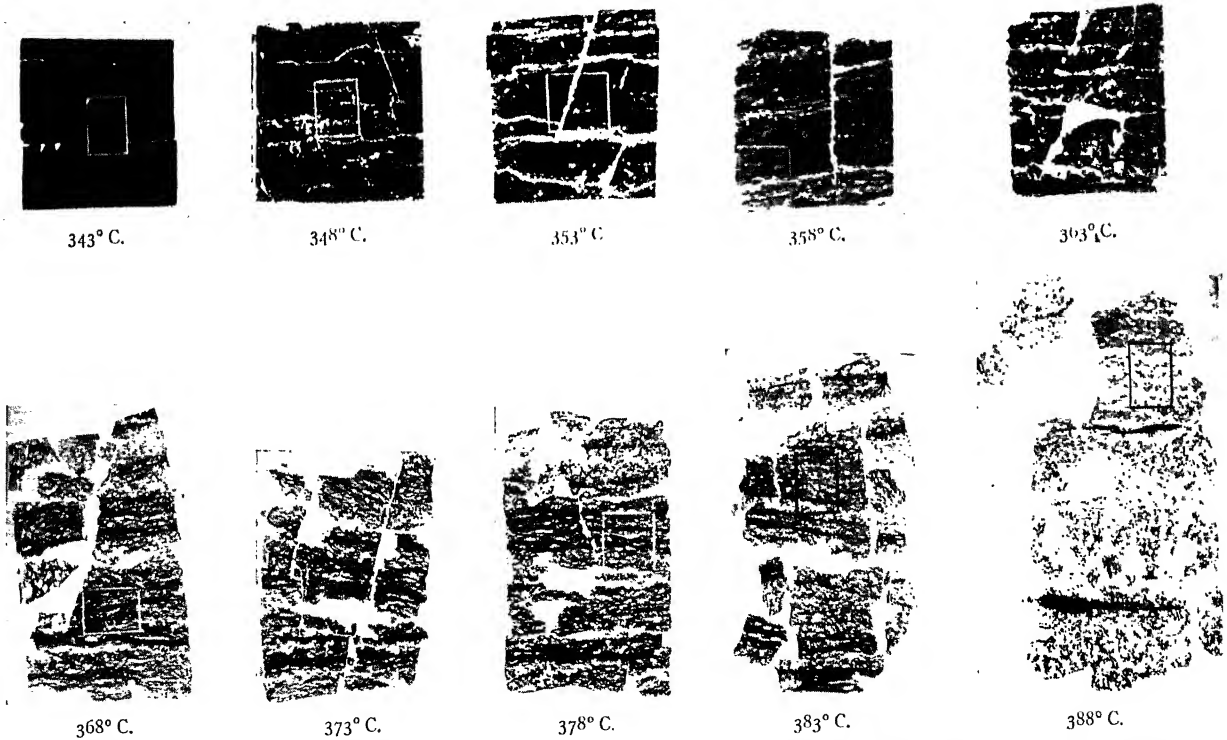


FIG. 94.—Rose Sections of 1-in. Cubes of Coal (times one) Heated to Different Temperatures. R.M. Parkgate Coal (×1). (See Fig. 95 for enlargements of rectangles marked.)

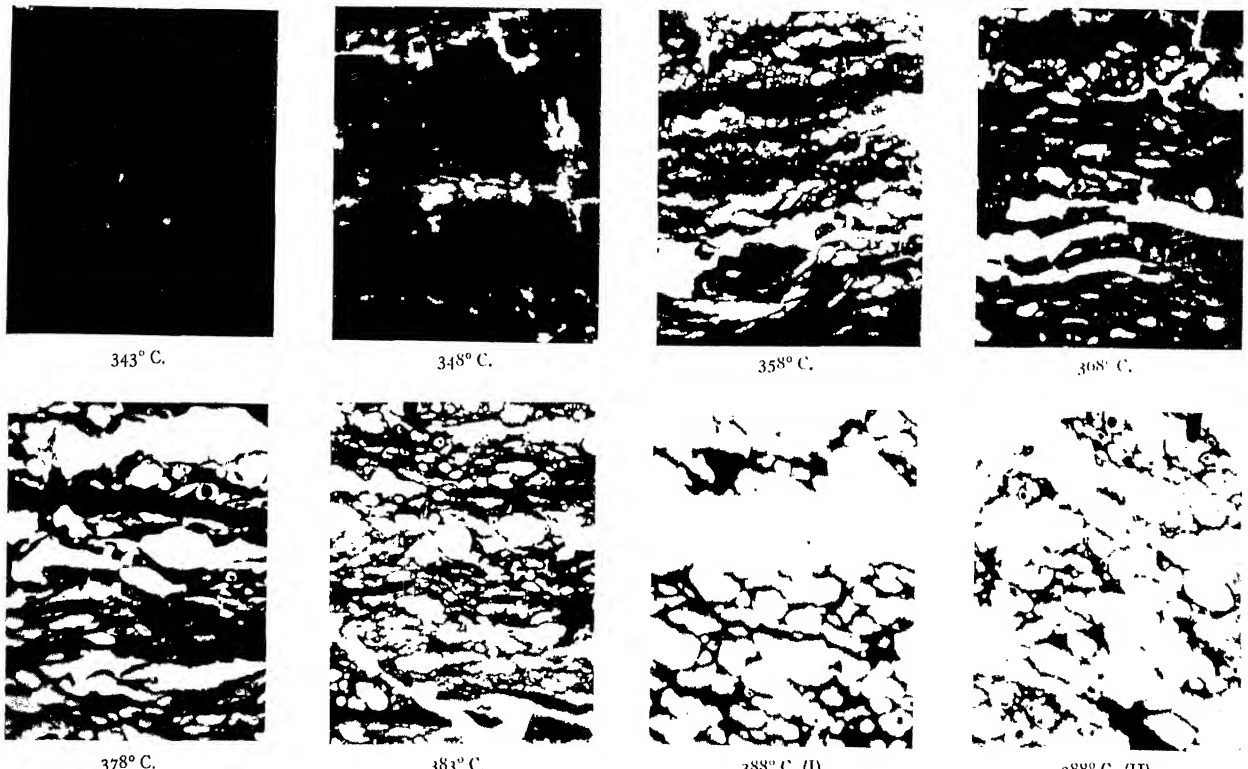


FIG. 95.—Rose Sections of 1-in. Cubes of Coal Heated to Different Temperatures. R.M. Parkgate Coal (×10). (See also Fig. 94.)

tray, so that no two pieces were touching, and heating them to 600° C. at a rate of 1° C. per minute in an inert atmosphere. Their volume was then measured by displacement of water in a volumenometer and the percentage swelling thus determined, with the following results :

Size of Coal, in.	1	$\frac{3}{8}$–$\frac{1}{4}$	$\frac{1}{4}$–$\frac{1}{8}$	$\frac{1}{8}$–$\frac{1}{20}$	$\frac{1}{20}$–$\frac{1}{40}$
Swelling, per cent.	130	100	115	110	120

The swelling was independent of the size of the original coal.

A single piece of R.M. Parkgate coal was also tested by the Sheffield Laboratory Coking Test for comparison with the usual test on a column of particles. The results were :

	Single Piece of Coal. No Load	Single Piece of Coal. Load 100 grammes	Column of Particles. Load 100 grammes
Temp. of Initial Contraction .	—	341° C.	342° C.
Temp. of Initial Expansion .	340° C.	368° C.	395° C.
Temp. of Final Expansion .	?	400° C.	412° C.
Contraction, per cent. .	nil	1	19
Expansion, per cent. .	200–350	317	130

The evolution of gases from lump and powdered coal (R.M. Parkgate) was compared by heating them in the Gray-King assay apparatus with a rate of rise of temperature of 1° C. per minute. The lump coal was in the form of prisms (two) $\frac{1}{4}$ by $\frac{1}{4}$ by 1 in. long, and the powdered coal was through 60-mesh. The results were almost identical, as is shown in Table XCVIII :

TABLE XCVIII—EVOLUTION OF GASES (C.C. PER GRAMME) FROM LUMP AND POWDERED COAL PER 10° C. RISE IN TEMPERATURE.

Range of Temp. ° C.	340–350	350–360	360–370	370–380	380–390	390–400	400–410	410–420	420–430	430–440
Lump coal . .	0·2	0·3	0·3	0·5	0·8	1·0	1·8	2·7	4·3	6·4
Powdered coal . .	0·1	0·3	0·4	0·4	0·7	0·9	2·0	3·1	4·3	5·3

In Fig. 96 the percentage changes of volume of a single piece of coal, both under no load and under a load of 100 grammes, and of columns of particles (through 60-mesh) under loads of 2 and 100 grammes, all heated with a rate of rise of temperature

of 1° C. per minute, are plotted against temperature. On the same graph appear data regarding the evolution of gas (which are the same for lump and powdered coal) and of oil.

The changes in dimensions of R.M. Parkgate coal when it is heated under different conditions are indicated in Table XCIX.

In all the tests a change occurs just above 340° C., there being a slight expansion of the single lump of coal under no load, but a

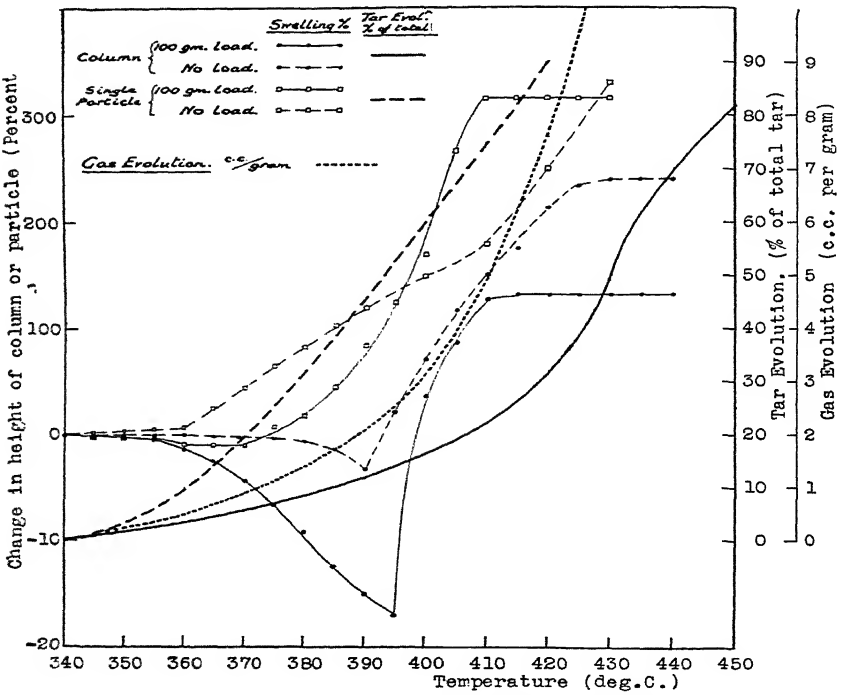


FIG. 96.—Comparison of (*a*) Changes in Volume, (*b*) Evolution of Gas and Oil from Single Lumps and Column of Particles at Different Temperatures. R.M. Parkgate Coal.

contraction of the column of particles. At about 365° C. the changes are accelerated, and over the range 365°–395° C. there is marked expansion of the single lump though contraction of the column of particles is continued. Between 395 and 410° C., expansion is marked both for the single lump and the column of particles, whilst between 410° and 430° C. expansion under load ceases.

It is evident that this coal begins to be plastic at about 340° C., but that the degree of plasticity is small until about 365° C. The relatively small change in the height of the column of particles over the range 365–395° C. shows the influence of surface phenomena,

the tendency of each individual particle to swell being counteracted by the ready release of gas from it. Above 395° C. the gas is evolved more rapidly than it can be released, and the column of particles begins to swell.

TABLE XCIX.—CHANGE IN HEIGHT OF R.M. PARKGATE COAL, PER CENT.

Range of Temp. ° C.	Single Lump (1-in. Cube)		Column of Particles (Through 60-mesh)	
	No Load	Load 100 gm.	Load 2 gm.	Load 100 gm.
345–365	+ 7	−0·9	−0·1	−2·4
365–395	+130	+128	−3·9	− 17
395–410	+ 30	+190	+130	−145
410–430	+150	nil	+ 88	+ 4

Fig. 96 illustrates a point which has been discussed earlier, namely, the more ready evolution of oils from a single lump than from a charge of powdered coal. No doubt oil is actually released most readily from the smallest particles, but is held on the surface of adjacent particles by adsorption. Such films of oil evolved below 340° C. have a "coalescing" effect on a charge of the powdered coal but do not cause it to become plastic, as witness the "coalescence indices" (see Fig. 93). The fact that changes in dimensions of the coal only become marked above 365° C. under each of the conditions of heating studied (Table XCIX) suggests that the same phenomena are concerned in each; that the contraction of a column of particles under load is due to the same physical condition of the coal as the expansion of a single lump, loaded or unloaded.

The films of oil on the particles in a charge of coal no doubt influence the extent to which the charge eventually swells or, if it does not swell, the extent to which contraction persists. But the dominant factor in the development of "primary" plasticity (in which each particle swells before the whole charge does) is the evolution of gas. This is shown in Fig. 97, which records the softening-points for columns of particles of different coals under a load of 100 grammes in the Sheffield Laboratory Coking Test. For coals of between 81–88 per cent. carbon and 5·25–5·45 per cent. hydrogen the softening-point is almost constant, though when the hydrogen-content is lower than 5·25 per cent. the softening-point is usually higher. When the carbon-content is less than 81 per cent. the softening-point may be as low as 275° C. It is only with such coals that the rate of evolution of gas below 340° C., under the conditions of test, is appreciable.

The close relationship between the softening-point as determined for a column of particles of coal and the temperature of initial swelling of a single lump has been followed by heating 1-in. cubes of different coals to temperatures near and above the softening-point. The results are given in Table C.

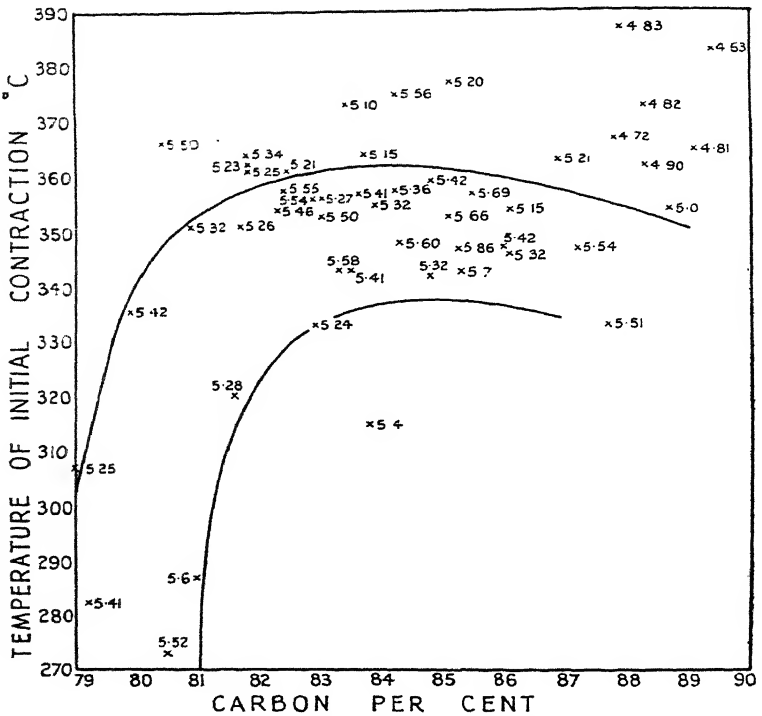


FIG. 97.—Softening Points of Coals in the Sheffield Laboratory Coking Test, Compared with Carbon Contents (Dry, Ash-free). The figures on the graph are hydrogen contents.

There is hardly any swelling of single lumps of coal at the softening-point as determined for a column of particles, but at the swelling-point of the latter, which is about 50° C. higher than the softening-point, the single lumps of coal swell considerably.

All coals except anthracites (and fusains) have been found to soften and contract in the Sheffield Laboratory Coking Test, and single lumps of all bituminous coals have been found to swell when the rate of rise of temperature is 1° C. per minute. (*Fuel in Science and Practice*, 1933, **12**, 185.)

The swelling of a lump of coal occurs for the most part at right angles to the bedding-plane, as is shown in Table CI, which records

the changes in volume of 1-in. cubes of R.M. Parkgate coal heated to different temperatures, together with their vertical and horizontal expansions. The vertical expansion is at right angles to the bedding-plane.

TABLE C.—SWELLING OF LUMPS OF COAL NEAR THEIR SOFTENING-POINTS.

Coal	Softening Point ° C. (Column of Particles)	Swelling of Lumps, per cent.			
		Temp. ° C.	Swelling, per cent.	Temp. ° C.	Swelling, per cent.
Silkstone (brights)	347	355	nil	405	75
Parkgate R (brights)	342	340	3	400	130
Parkgate A (brights)	357	360	40	395	160
Barnsley (brights)	364	355	1	400	130
Swallow Wood (brights)	364	360	10	395	90
Parkgate A (hards)	385	370	nil	400	25
Barnsley (hards)	383	370	4	405	35
Swallow Wood (hards)	372	360	nil	410	19

TABLE CI.—THE EXPANSION OF 1-IN. CUBES OF R.M. PARKGATE COAL (PER CENT.).

Temp. of Heating ° C.	Increase in volume	Increase in height (vertical plane)	Increase in width (horizontal plane)
330	nil	nil	nil
340	3	4	−0·5
350	3	3·5	−1·5
360	60	3·7	8
370	76	38	12
380	94	67	7
390	100	80	6
400	148	52	28
410	174	40	24
420	250	84	41
430	340	97	64

A different cube of coal was used for heating to each temperature, which no doubt accounts for the irregularities in some of the values recorded in Table CI. A marked increase in the swelling is observed in a vertical plane at 370° C. and in a horizontal plane at 400° C., the latter corresponding with a considerable increase in the volume (see Figs. 94 and 95). No doubt the expansion in a horizontal plane (*i.e.* parallel to the bedding-plane), which completely destroys the original shapes of the particles, accounts for the initial swelling of a column of particles of bright coal which occurs over about the same temperature-range. Single lumps of durain, on the other

hand, do not expand considerably, parallel to the bedding-plane, and columns of particles of durain do not swell.

The swelling in a horizontal plane with cubes of R.M. Parkgate coal increases at a temperature corresponding with that at which a column of particles of the same coal swells (395° C.), as is shown in Table CII, which records also the dimensions of the "pressed" cube of Fig. 98.

Not Pressed Pressed

FIG. 98.—Rose Sections of 1-in. Cubes of Coal Heated to 380° C. Pressed and not Pressed. R.M. Parkgate Coal.

TABLE CII.—THE EXPANSION OF CUBES OF R.M. PARKGATE COAL.

Temp. ° C.	Percentage increase in :		
	Volume	Height	Width
340	3	4	−0·5
360	100	88	8
380	124	110	10
400	212	100	27
380*	38	31	2·5

* Pressed whilst hot.

No doubt external pressure is mutually applied by the swelling of adjacent particles in a column and prevents appreciable expansion of the column below the temperature of initial expansion.

The fact that a 1-in. cube of coal heated to a temperature of about 370° C. can swell without cracking (Figs. 94 and 95) shows that it is plastic at that temperature. The plasticity can be demonstrated by applying pressure to the cube of heated coal. A cube of R.M. Parkgate coal heated to 360° C. (when a 100 per cent. increase in

volume occurred) was pressed by hand, when it contracted but broke into pieces as the pressure continued. A similar cube heated to 380° C. (when the increase in volume was 125 per cent.) contracted under pressure without breaking. Fig. 98 shows the appearance of such heated cubes of coal before and after being subjected to pressure. In the example given, the pressed cube was about 38

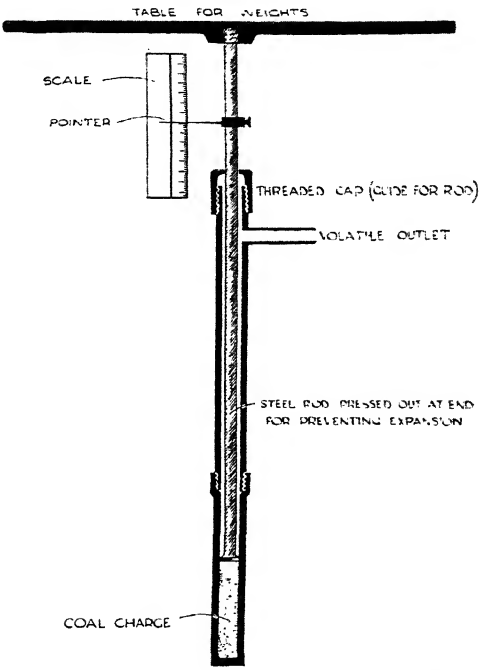


FIG. 99.—Apparatus used to Measure the Effect of Different Loads on the Swelling of Coal.

per cent. greater in volume than the unheated cube. The pore-structure has been retained but the pores are elongated parallel to the bedding-plane.

A charge of particles, as such, may not be uniformly plastic although in each individual particle plasticity has been developed. At the outset of the plasticity of the particles in a charge there is about 33 per cent. free space. This free space must be filled before the charge attains "secondary" plasticity, when it swells as though it were a single lump. At this stage the boundaries between single particles have disappeared and pores, which are large in size in relation to that of the particles, are produced by enclosure of the free spaces between the particles.

The softening-points heretofore recorded have been for columns of particles under a load of 100 grammes. At higher pressures, however, the softening-point (as indicated by a contraction in volume) may be lower. The effect of pressure on the temperature of initial contraction has been studied with the apparatus illustrated

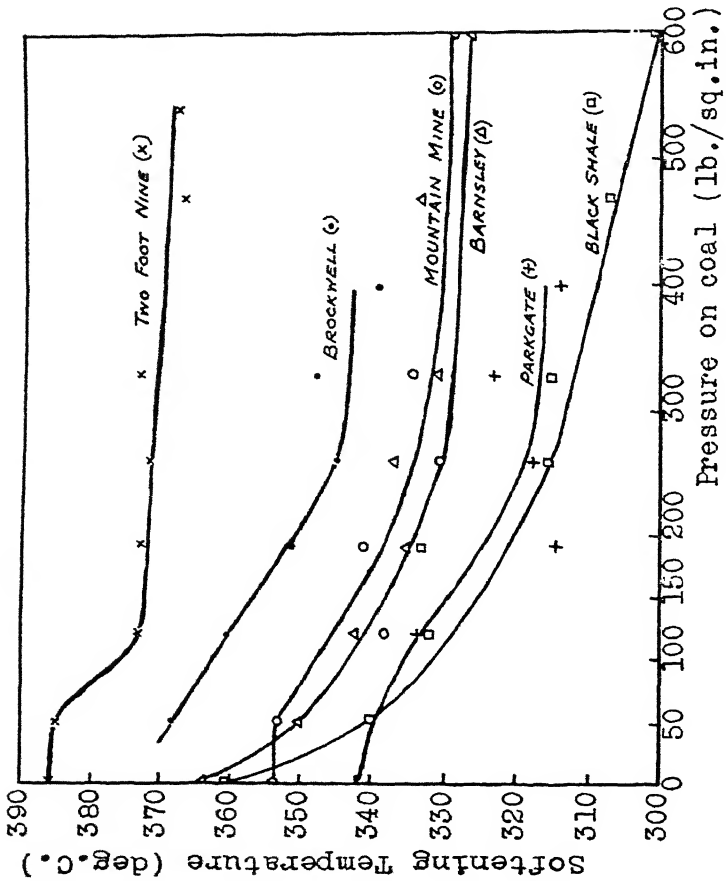


FIG. Effi

in Fig. 99, which is self-explanatory. The tube was of steel and about ⅜-in. in diameter, whilst the steel plunger had a clearance of about $\frac{1}{64}$-in. The results of several series of tests at different pressures are recorded in Fig. 100. The effect of pressure on the softening-point varies somewhat with the coal but, in general, is of no moment up to 50 lb. per sq. in. Between 50 and 200 lb.

per sq. in. increased pressure lowers the softening-point, which then remains nearly constant up to a pressure of 600 lb. per sq. in.

The fact that in these experiments no change occurred in the softening-point (temperature of initial contraction) with pressures up to 50 lb. per sq. in. shows that the values recorded for different coals, under a load of 100 grammes, in the Sheffield Laboratory Coking Test can be taken as reasonably close to the temperature of primary plasticity in a coke-oven, since the mean external load is quite small in coke-oven practice (about 2 lb. per sq. in.). The decrease in the softening-point as the pressure is increased above 50 lb. per sq. in. is interesting in connexion with the evidence already adduced that the initial "softening" of a column of particles is due to the swelling of individual particles which, in turn, is due to the evolution of gas.

Tests made with coals that were prevented from swelling, described in a later chapter (p. 300), showed that Parkgate coal remained plastic up to 480° C., Silkstone to 510° C. and Two Foot Nine to 520° C. or higher. For two Durham coals it was not possible to determine at what temperature they ceased to be plastic, for the reason that the gas retained by them whilst plastic created a higher pressure than it was possible to apply externally with the apparatus available.

In general, when swelling of the coal is prevented, plasticity seems to persist to a higher temperature the better the coking quality of the coal. This is in accord with practical experience, inasmuch as cokes from the better coking coals are the more "fused" and their real specific gravities are the higher.

TABLE CIII.—COMPARISON OF LOWEST TEMPERATURES OF INITIAL CONTRACTION OF COALS (UNDER PRESSURE) WITH THEIR ACTIVE DECOMPOSITION-POINTS.

Coal	Carbon, per cent., on dry, ash-free basis	Lowest Temp. of Initial Contraction, ° C.	Coal	Carbon, per cent., on dry, ash-free basis	Active Decomposition-Point, ° C.
Two Foot Nine	89·5	370	Two Foot Nine	90	370
Brockwell .	88	345	Busty . .	88	340
Mountain Mine	86	330	Silkstone .	84	330
Silkstone .	86	330	Parkgate .	83·5	330
Parkgate .	85	315	Wigan Six Foot	83	320
Barnsley .	84	330	Barnsley .	82	310
Black Shale .	82	300	Thick . .	81	300

Whilst the relationship between the temperature of initial plasticity and the temperature of initial rapid evolution of gas suggests that a high internal gas-pressure conduces to plasticity,

it is well-known that a high external pressure, as used in the manufacture of "pure coal" briquettes, can render coal plastic at a low temperature. It may well be, therefore, that the combined effects of the internal and the external pressure in the experiments

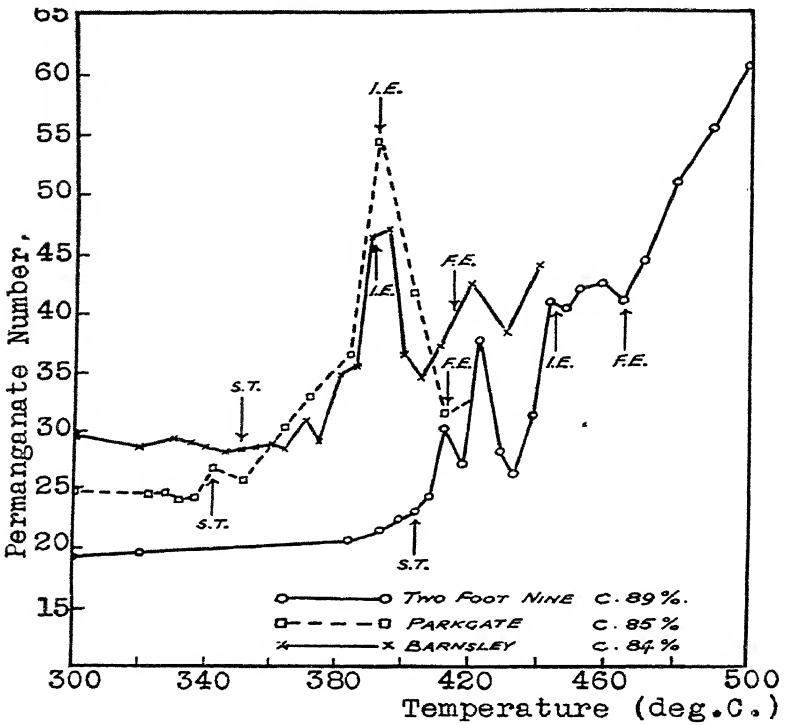
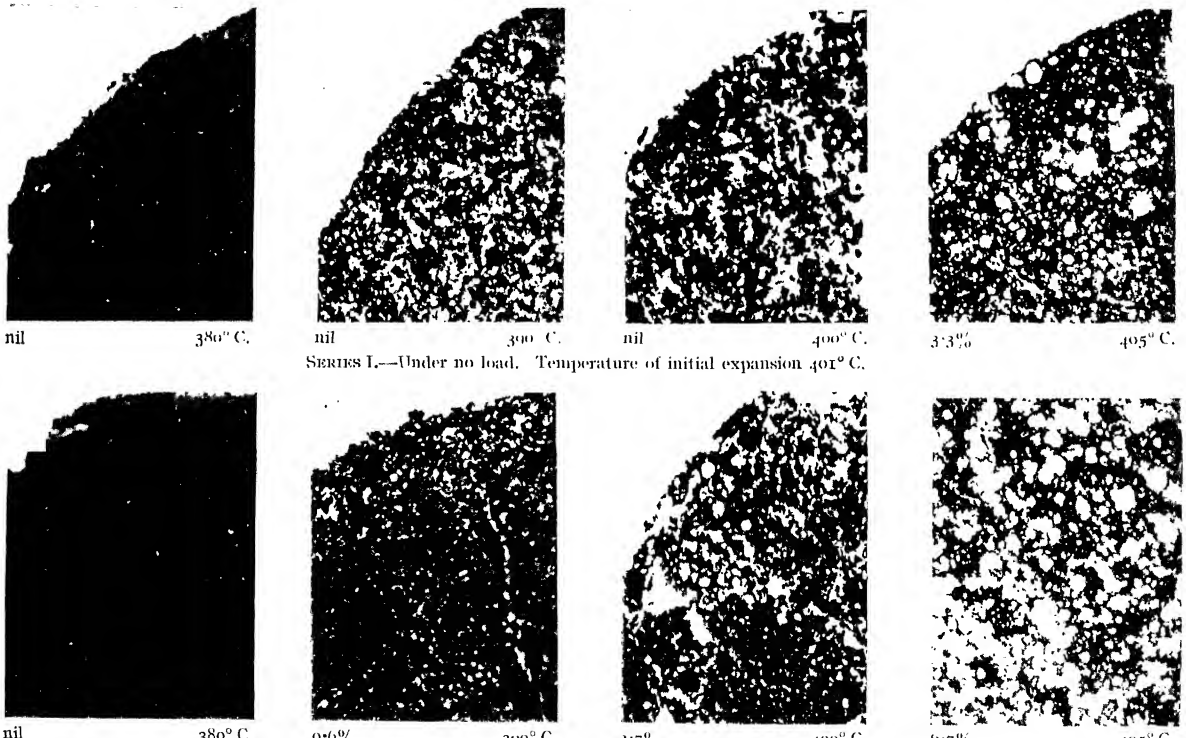


FIG. 101.—The Increased Reactivity of Coals at their Softening Temperatures (S.T.). (With some Coals, other Critical Points occur at the Temperatures of Initial Expansion (I.E.) and of Final Expansion (F.E.)

just described account for the reduction in the temperature of initial contraction (*i.e.* of the softening-point) as the external pressure is raised from 50 to 200 lb. per sq. in. It is significant, in this connexion, that the lowest temperatures of initial contraction of coals under pressure correspond roughly with their "active decomposition-points" as determined by Holroyd and Wheeler, as is shown in Table CIII.*

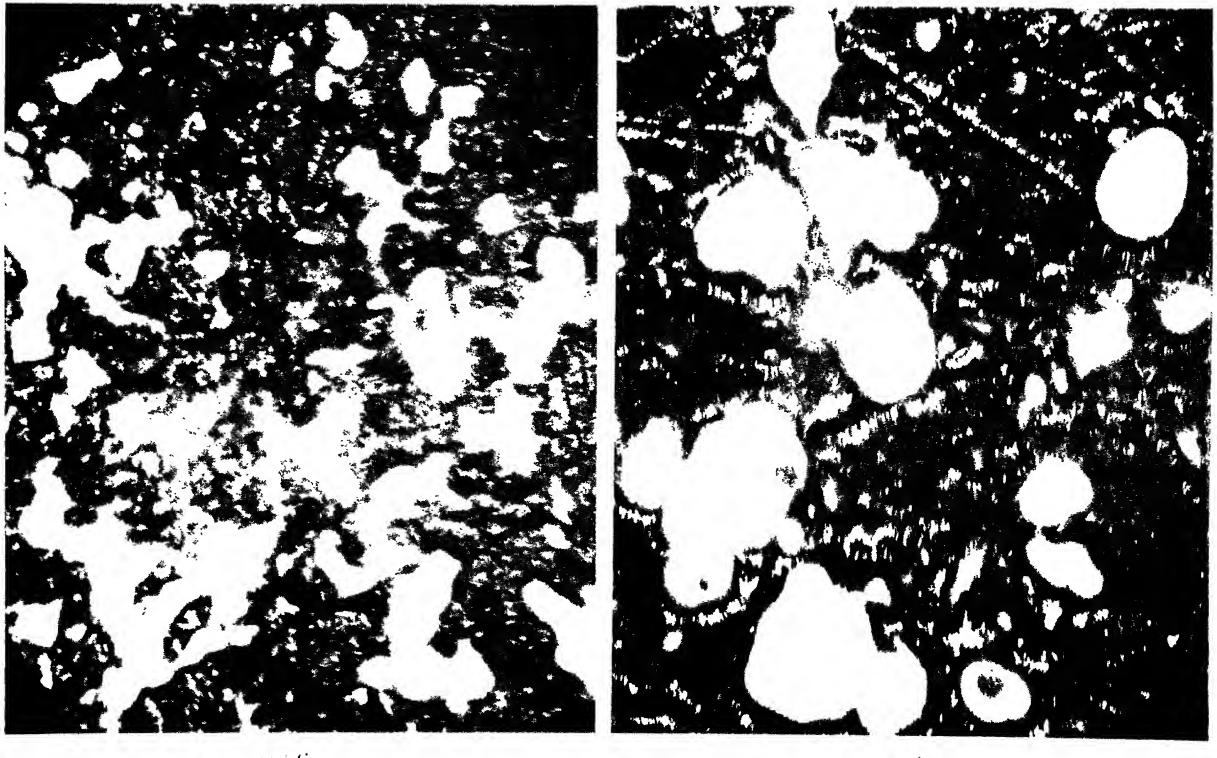
* Comparison should be made between coals of the same carbon content. The coals of very low rank, such as the three which soften below 300° C. in Fig. 97, undergo partial decomposition (of external groupings) below the active decomposition-temperature. Such coals, although they soften below their active decomposition-temperatures, do not do so before relatively large volumes of gas have been released.



SERIES I.—Under no load. Temperature of initial expansion 401° C.

SERIES II.—Under load of 100 grm. Temperature of initial expansion 398° C.

FIG. 102.—The Formation of Closed Pores at the Temperature of Initial Expansion R.M. Parkgate Coal (×10) (see also Fig. 103.)



400° C. 405° C.

FIG. 103.—The Formation of Closed Pores at the Temperature of Initial Expansion. R.M. Parkgate Coal (× 100), Series I. Under no load. Temperature of Initial Expansion 401° C. (Cf. Fig. 102).

This relationship between the lowest temperature of initial contraction of a coal under load and its active decomposition-point—which, incidentally, negatives the idea of there being coals which "melt" before they decompose, decomposition and softening being cause and effect—suggested another way of demonstrating the dependence of the primary plasticity of coal on the evolution of gas. Holroyd and Wheeler showed that immediately after the active decomposition-point of a coal had been passed its solid residue increased in reactivity, *e.g.* towards oxygen or oxidising agents (*see* also Heathcoat, *Fuel in Science and Practice*, 1933, **12**, 8). Samples of three representative coking coals were therefore heated, with a rate of rise of temperature of 1° C. per minute, to temperatures below and above their softening-temperatures, and the reactivities of the residues towards alkaline potassium permanganate were then determined. The results have been recorded graphically in Fig. 101.

It will be seen that for each coal the softening-point nearly coincides with a sharp rise in the reactivity of the solid residue.

The Initial Expansion of Columns of Particles

The temperature of initial expansion of a column of particles is the most clearly-marked of any of the significant temperatures recorded during the Sheffield Laboratory Coking Test. For a given rate of heating, it is not appreciably altered by the application of external pressure above about 2-lb. per sq. in., as is shown by the records in Table CIV for through 60-mesh R.M. Parkgate coal heated with a rate of rise of temperature of 1° C. per minute.

Table CIV.—The Effect of Pressure on the Temperature of Initial Expansion of a Column of Particles of Coal (R.M. Parkgate).

	Pressure on Coal, lb. per sq. in. :					
	2	50	120	190	260	330
Temp. of Initial Expansion, ° C.	388	389	387	389	387	387
Temp. of Final Expansion, ° C.	408	414	400	403	398	390
Expansion, per cent. . .	77	64	25	22	5	2

Charges of through 60-mesh R.M. Parkgate coal were heated in the Sheffield Laboratory Coking Test at temperatures below and above the temperature of initial expansion and the structures of the residues then examined under a magnification of 10 diameters. Photographs of typical structures, with the pores infilled as far as possible with plaster of Paris, are reproduced in Fig. 102 and at 100 diameters magnification in Fig. 103. It is evident from these

photographs that, at the temperature of initial expansion, the formation of pores throughout the mass, and not only in each individual particle, has begun. No doubt there are pores also in each, or some, of the particles, but they are too small to be infilled with plaster of Paris and thus revealed. The pores formed throughout the mass at the temperature of initial expansion are large in relation to the size of the particles. For example, in the product obtained at 400° C. (*i.e.* 5° C. above the temperature of initial expansion) there are many pores over 0·01 in. in diameter, whereas the largest particle of coal is 0·008 in. in diameter. Determinations of the proportions of closed and open pores in the different products gave the results recorded in Table CV.

TABLE CV.—THE FORMATION OF PORES AT OR NEAR THE TEMPERATURE OF INITIAL EXPANSION. (R.M. PARKGATE COAL).

Temp. ° C.	Under No Load (Temp. of Initial Expansion 401° C.)			Under Load of 100 Grammes (Temp. of Initial Expansion 398° C.)		
	Closed Pores, per cent.	Open Pores, per cent.	Real Specific Gravity	Closed Pores, per cent.	Open Pores, per cent.	Real Specific Gravity
350	nil	—	1·30	nil	—	1·29
300	nil	32	1·31	0·6	24	1·32
400	nil	26	1·32	2·7	12	1·32
405	3·3	14	1·31	6·7	25	1·33

The product obtained at 405° C. under a load of 100 grammes was highly cellular in structure, although the proportion of closed pores was small.

The pores are clearly defined and spherical at a temperature 4° C. above the critical temperature, but below that temperature they are of irregular shape and show no signs of the effect of gas-pressure. Since the proportion of closed pores is small, it is evident that the sequence of events is the closing of irregularly shaped cells, their distension by gas (giving them a spherical shape), and their subsequent disruption. It is impossible to distinguish, quantitatively, between open pores which have been formed according to this sequence, and other "open pores" which are, in fact, irregularly shaped voids, but Figs. 102 and 103 shows clearly the remarkable change in the character of the pores that occurs at the temperature of initial expansion.

At the temperature of initial expansion of a column of particles, the external surfaces of the particles coalesce and the "primary plasticity" of the individual particles gives place to the "secondary

plasticity" of the mass as a whole. In the state of primary plasticity, the aggregate swelling of the particles of coal is limited to about 33 per cent., equivalent to the amount of void space between the particles, and gas evolved from individual particles can escape through the voids. In the state of secondary plasticity, all the voids have been closed and the gas evolved has to force its way under pressure through a plastic mass which, in consequence, swells as a whole.

Summary

The softening-point (temperature of initial contraction) of a column of particles has been related to the decomposition-point of the ulmins, at which gas and oils are evolved freely. Below the softening-point, however, oils present in the coal in a loosely-bound form, as hydrocarbons and resins, are distilled to the surfaces of the particles of coal and are adsorbed there, where they act as an adhesive to sinter the particles together. Above the softening-point, each particle of coal begins to swell, the initial stage being marked by the formation at the bedding-planes of minute cracks which apparently become sealed by adsorbed oil and distended by gas.

The application of a high gas-pressure at a time when the ulmin complex is disrupting induces "surface flow" at so many of the rudimentary pores that each coal particle is plastic, though the plasticity is local ("primary" plasticity). As the temperature rises, the swelling of each particle brings plastic surfaces into contact and coalescence occurs. Finally, at the temperature of initial expansion, the original void spaces between the particles become enclosed by plastic material and the escape of gas is restricted; the column of particles then swells as a whole and the plasticity, no longer local, becomes "secondary" plasticity.

Foxwell has implied that the temperature at which a charge of particles of coal becomes impermeable to gas (*i.e.* our "temperature of initial expansion") is the temperature of "initial plasticity" (*Fuel in Science and Practice*, 1924, 3, 122), but this implication obviously requires qualification.

CHAPTER XVI

THE PLASTICITY OF COAL AND THE FORMATION OF SEMI-COKE

It has been shown how individual particles of a coking coal in a column of particles become plastic between the temperatures of softening and of initial expansion of the column, and this temperature-range has been termed that of "primary plasticity." At the temperature of initial expansion, a stage referred to as marking the beginning of "secondary plasticity," the column of particles becomes a single coherent structure which is uniformly plastic. Above the temperature of initial expansion (which, with a rate of heating of 1° C. per minute, occurs at about 400° C. unless the hydrogen-content of the coal is low), the column continues to swell until a temperature of about 420° C. is reached. The coal remains plastic, however, after swelling has ceased. Experiments, now to be described, have been made to determine how long, after swelling has ceased, plasticity persists.

The Determination of the Duration of Plasticity of Coal Free to Expand

When carrying out the Sheffield Laboratory Coking Test (see p. 272), the weighted plunger was clamped after the maximum degree of swelling had been attained and released after a measured interval of time, whilst the temperature of the coal continued to rise at a rate of 1° C. per minute. In a series of such tests, the weighted plunger was released after successively longer intervals of time until the column of coal was no longer penetrated by it. Details of tests with one coal (R.M. Parkgate) are recorded in Table CVI. The temperature of final expansion of this was 413° C., the percentage of expansion at that temperature being 126.

Plasticity ceased between 433° and 443° C., so that the "solidification-temperature" of this coal under the conditions of test can be taken as about 438° C. The solidification-temperatures of a number of coals were determined in the same manner, with the results given in Table CVII.

It will be seen that, unless the amount of expansion at the temperature of final expansion exceeded about 125 per cent., the solidification-temperature was no greater than the temperature of final expansion.

TABLE CVI.—THE DETERMINATION OF THE DURATION OF PLASTICITY OF A COAL (R.M. PARKGATE) FREE TO EXPAND.

Time after Final Expansion, min.	Temp. ° C.	Contraction on Release of Plunger, per cent.
Nil	413	100
10	423	25
20	433	12·5
30	443	nil

TABLE CVII.—SOLIDIFICATION-TEMPERATURES OF COALS FREE TO EXPAND.

Coal	Temp. of Final Expansion, ° C.	Expansion, per cent.	Solidification-Temp., ° C.
R.M. Parkgate	413	126	438
Black Shale	403	168	429
Brockwell	428	184	443
Swallow Wood T.	423	193	428
Tinsley coking slack	427	126	427
Swallow Wood A.	413	89	413
Silkstone	428	80	428
Tupton	416	93	416
Wigan 6 ft.	427	54	427
Deep Soft	408*	nil	408
Shafton	413*	nil	413

* Non-swelling coals: the temperatures given for them are temperatures of final contraction.

In the Sheffield Laboratory Coking Test, a good coking coal swells rapidly just above the temperature of initial expansion, but, later, the degree of swelling in unit time decreases with increase in temperature, as is shown in Table CVIII, for typical Durham, Welsh and Yorkshire coking coals.

TABLE CVIII.—SWELLING, PER CENT., FOR SUCCESSIVE 5° C. RISES IN TEMPERATURE ABOVE TEMPERATURE OF INITIAL EXPANSION.

Coal	Percentage Swelling per 5° C.						Total
Two Foot Nine (S. Wales)	16	60	48	17	5	—	146
Busty (Durham)	15	46	48	51	23	2	205
Victoria (Durham)	46	36	24	20	1	—	137
Silkstone (S. Yorkshire)	22	38	25	12	2	—	160
Parkgate (S. Yorkshire)	29	42	42	17	—	—	130

It is unlikely that the reduced rate of swelling as the temperature rises should be due to a decrease in viscosity of the mass of coal (*i.e.* to an increase of plasticity) for, by the time that a decrease in the rate of swelling has become apparent, the column of powdered coal has distended to about twice its original length. A decrease in viscosity, providing greater ease of escape of gas, would cause the distended mass to subside, whereas it continues to expand, though at a slower rate.

It is probable that there is a continuous decrease of plasticity, due to progressive solidification of the mass. The expansion of the coal to twice its original volume provides within the porous mass an extensive surface at which the molecules, being bound only on one side remote from the surface, have a degree of freedom associated rather with the liquid than the solid state.* Above the temperature of initial expansion of the coal, the rate of evolution of gas increases rapidly, and the mechanical effect of this escape of gas no doubt accelerates such reactions and may cause rearrangement of the molecules. The rupture of the less stable groupings and their expulsion as "volatile matter" also encourages the attainment of a relatively stable form, with progressive solidification, as in the "vitreous state" postulated by Beilby.†

It is difficult to measure the relative degrees of plasticity of coal during the formation of coke. Even when the kind of coal, its rate of heating and its degree of freedom to expand during heating are standardised, there remains the difficulty that the plastic coal undergoes progressive decomposition so that new substances are being continuously formed. Perhaps the most convincing explanation of the behaviour of coal in the plastic state is that which takes account of the manner of evolution of volatile matter.

TABLE CIX.—RATIOS FOR THE EVOLUTION OF GAS OVER RANGES OF 10° C. FOR DIFFERENT COALS.

Coal	$\frac{410–420° C.}{400–410° C.}$	$\frac{420–430° C.}{400–410° C.}$
Brockwell . . .	1·7	3·0
Busty . . .	1·9	4·0
Silkstone . . .	1·4	2·3
Parkgate . . .	2·0	2·6

Allinson and Mott (*Fuel in Science and Practice*, 1933, **12**, 262) have demonstrated that during the plastic stage of coal the rate of

* In a liquid, below its surface, each molecule is influenced by the attraction of molecules on all sides, whereas at the surface the molecules are only influenced by other molecules below the surface. The molecules at the surface of liquids (and of solids) have, in consequence, special properties; such, for example, as provide the phenomena of surface tension.

† G. Beilby, "Aggregation and Flow of Solids," London, 1921.

evolution of gas increases. Up to the temperature of initial expansion (which, as previously stated, is usually about 400° C. with the standard rate of heating in the Sheffield Laboratory Coking Test), the evolution of gas is only about 3 c.c. per gramme from a good coking coal, but this rate may be doubled as the temperature rises 10° C. and trebled during a succeeding rise of 10° C. Results for two Durham and two Yorkshire coking coals are given in Table CIX by comparing the production of gas per gramme of coal over different temperature ranges.

The rate of evolution of gas continues to increase over each 10° C. rise in temperature up to about 460° C., as is shown by the following results for Parkgate coal:

Temp. °C. .	390	400	410	420	430	440	450	460	470	480	490	500
Gas, c.c. per gramme .	—	0·9	1·7	2·6	4·0	5·0	5·7	6·5	6·4	5·8	5·4	5·8

It will be shown later how great is the magnitude of the pressure of gas within the swelling coal. At present it is sufficient to note that the coal continues to swell until the internal gas-pressure within the pores balances the external resistance. We assume that the plasticity of a coal is at its maximum during the early stages of swelling in the Sheffield Laboratory Coking Test, and when the rate of increase in volume per 5-minutes heating (5° C.) begins to fall off we assume that the plasticity of the coal begins to decrease. The tendency that there then is for the coal to solidify does not prevent further expansion, because an increasing rate of production of gas, with consequent increasing internal pressure, overcomes the resistance of the gradually solidifying walls of the pores. As solidification proceeds, the pores, after they burst to release gas, close or reform with increasing difficulty, owing to the increasing viscosity of their walls, until finally most of them remain open. The escape of gas from the decomposing coal is then greatly facilitated and there is no further swelling, since the internal pressure falls, although the mass of the coal is still plastic. Under the conditions of the test, the weighted plunger can now penetrate the coal until such time as actual solidification of the charge arrests it. The charge of coal does not subside as a whole under the weight of the plunger, showing that, until solidification actually occurs, there is still some closure of pores and retention of internal gas-pressure.

The Influence of External Pressure on the Plasticity of Coal.

The deductions to be drawn regarding the plasticity of coal from the results of tests on charges of coal which are free to expand do not necessarily apply to charges which are prevented from expanding, as in the coke-oven. In a coke-oven the isothermal lines are parallel to the walls, and the plastic layers at any given

period during coking are vertical bands, each bounded on one side by uncarbonised coal and on the other by a "carbonised" mass, which ranges from semi-coke near the plastic layer to fully-formed coke near the wall. Within the plastic layers the tendency for any given particles of coal to expand (beyond the range given by the void spaces in the charge) is checked by the same tendency of superincumbent particles, as is shown diagrammatically in Fig. 104. Only in the uppermost layer of the charge, therefore, is there full freedom for upward movement, and the "spongy" coke formed in consequence is but a thin layer on the top of the charge. Upward movement of a charge of coal in an oven is small, and, broadly

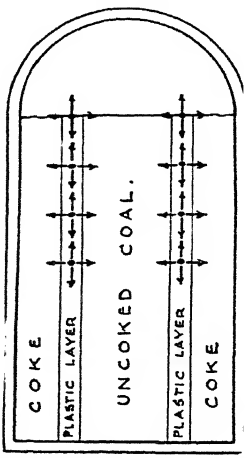


FIG. 104.—Diagrammatic Representation of the Neutralisation of the Vertical Components of Swelling Pressure in a Coke Oven.

speaking, the formation of coke can be considered to be taking place at a constant volume. A "swelling pressure" is thus developed which may affect the plasticity of the coal. We have therefore made tests to determine this swelling pressure and measure its effects.

A modification of the apparatus for the Sheffield Laboratory Coking Test was used for the tests. The retort consisted (see Fig. 99) of a tube of mild steel, $\frac{3}{8}$-in. in internal diameter with walls $\frac{1}{8}$-in. thick, comprising two portions screwed together to enable coked residues to be removed easily. A plunger of mild steel, $\frac{1}{4}$-in. in diameter, expanded at its lower end to provide a clearance of $\frac{1}{64}$-in., fitted into the retort. The upper part of the plunger passed through a collar screwed on the top of the retort and carried a platform upon which weights could be placed.

The tests were carried out in the same way as in the Sheffield Laboratory Coking Test except that, in a series of tests with a

particular coal, the weight on the platform was varied. The results of such a series of tests are given in Table CX.

TABLE CX.—THE EFFECT OF PRESSURE ON THE SOFTENING AND SWELLING OF COAL. (R.M. PARKGATE COAL.)

Pressure, lb. per sq. in.	Temp. (° C.) of: Initial Contraction	Initial Expansion	Final Expansion	Contraction, per cent.	Expansion, per cent.
50	350	400	425	26	55
120	342	400	411	26	24
190	335	400	410	35	15
260	337	401	410	35	20
330	335	401	408	36	15
470	334	399	407	35	10
610	327	402	404	36	2
750	325	400*	—	33	nil

* Final contraction, coal did not expand.

A pressure of about 750 lb. per sq. in. (50 atm.) was thus required to prevent the expansion of this coal during heating. With other coking coals, the pressure necessary to prevent their expansion during heating, under the conditions described, ranged from 400 to 750 lb. per sq. in.

Such high pressures as these give some idea of the magnitude of the forces operating during the formation of coke. In the tests, the gas evolved was able to escape readily around the plunger, which moved upwards smoothly during expansion of the charge, showing that the gas in the mass of plastic coal beneath the plunger exerted a steady pressure slightly greater than the external pressure. In this respect, the plasticity of coal differs markedly from the plasticity of pitch, for example, which produces a negligible internal gas-pressure when it is heated.

An attempt was now made to determine the temperature at which coal that was prevented from expanding ceased to be plastic, in the same manner as in the tests, described earlier, on charges of coal that were free to expand. To avoid complication due to the preliminary contraction of the charge under a heavy load during the period of "primary plasticity" (which does not occur in a coke-oven), the plunger was clamped until the coal began to expand. The plunger was then loaded, first with a sufficient weight to stop the expansion and then, after the expansion-pressure had ceased, with sufficient to cause the plunger to begin to sink into the charge of coal, if this were possible.

Experimental difficulties prevented satisfactory determinations of such "solidification-temperatures" with coals of high carbon-content. Results for five coals are recorded graphically in Fig. 105. For Parkgate coal, expansion began at about the same temperature

as in the normal Sheffield Laboratory Coking Test and the pressure required to prevent expansion increased regularly up to about 250 lb. per sq. in. as the temperature rose to 415°. Over the range 415–430° C. the pressure necessary to prevent expansion remained steady, but thereafter it increased rapidly until at 440° C. it was 600 lb. per sq. in. and the plunger began to sink into the charge of coal. Over the range 440–460° C., the minimum pressure necessary to cause the plunger to sink into the charge fell slowly to 100 lb. per sq. in., but thereafter rose rapidly to 600 lb. per sq. in. when the temperature was 480° C. Similar results were obtained with

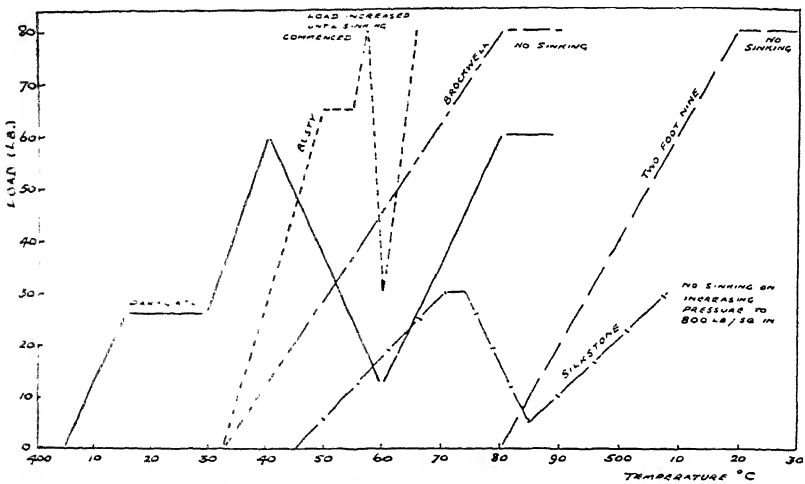


FIG. 105.—The Load required to Prevent Swelling and to Cause the Plunger to begin to sink through the Charge. Various Coals.

Silkstone coal, and the "solidification-temperatures" of these two coals when heated at constant volume can be taken to be about 480° C. and 510° C.

For one Welsh and two Durham coking coals, simpler types of graphs were obtained. For Busty coal, the pressure required to prevent expansion increased to 650 lb. per sq. in. up to 450° C. and this pressure was sufficient whilst the temperature rose 5° C., but there was no tendency at this stage for the plunger to sink into the charge of coal, as with the Parkgate and Silkstone coals. Not until the pressure was increased to 800 lb. per sq. in. did the plunger begin to sink. As the temperature rose a further 5° C. the pressure required became less, only to rise rapidly again beyond 800 lb. per sq. in. (the highest pressure that could conveniently be applied) after a further rise of temperature of 5° C. Neither for the Brockwell nor the Two Foot Nine coal could the "solidification-temperature" be determined.

For two coals, therefore, of 85 and 86 per cent. carbon-content, the tests determined the "solidification-temperature" at constant volume, but for coals of higher carbon-content, 87 to 89 per cent., the tests failed because, with the apparatus available, sufficiently high pressures could not be applied. This fact is in itself important as showing that, in coke-oven practice, the better coking coals must exert higher pressures within the oven.*

Foxwell (*Fuel in Science and Practice*, 1932, **11**, 370) obtained similar results in tests in which he charged coal into the middle portion of a steel tube of 1-in. bore, and prevented it from expanding when it was heated by filling the remainder of the tube with crushed firebrick. The gas evolved was collected and measured. A second tube containing another charge of the same coal mixed with crushed coke was heated simultaneously in the same furnace and the gas evolved from this charge was also measured. The presence of the coke in the second charge prevented the particles of heated coal from coalescing so that there was no restriction of the evolution of gas and a comparison of the volumes evolved from the two charges showed the amount of gas imprisoned in the plastic coal. A separate test was made to determine the amount of water-vapour evolved, and the pressure in the charge of coal at any temperature was calculated by dividing the volume of gas and water-vapour imprisoned in the plastic coal by the volumes of free space in the charge. The pressures thus calculated varied between 285 and 2,000 lb. per sq. in. (20 and 140 atm.), and were greater the faster the rate of heating and the greater the bulk-density of the charge. Under comparable conditions, the higher pressures were usually obtained with the better coking coals.

Methods of Measuring the Plasticity of Coal

Several methods have been devised to measure the plasticity and the plastic range of coal. J. D. Davis (*Journ. Ind. Eng. Chem.* [*Anal.*] 1931, **3**, 343) measured the resistance of rabble arms moving through coal loosely charged in a horizontal rotary retort, and therefore free to swell. A number of results with the apparatus are recorded in U.S. Bureau of Mines Monograph 5, 1934 ("*Gas-, Coke-, and By-Product-Making Properties of American Coals and their Determination*," by A. C. Fieldner and J. D. Davis). The rate of heating through the plastic stage was 3·4° C. per min. Some of the results are recorded in Table CXI.

In the Damm-Agde test a briquette of coal was heated at a rate of 4·2° C. per min., under a load of 10 lb. per sq. in., and the temperatures at which contraction and expansion began were measured. The temperature of final expansion could only be measured for weakly-swelling coals owing to the range of the gauge

* The internal swelling pressure measured in this test should be distinguished from the residual swelling tests of Damm, Nedelmann or Koppers, in which the coal is not uniformly at the same temperature.

TABLE CXI.—U.S. BUREAU OF MINES INVESTIGATION OF PLASTIC PROPERTIES OF COAL.

Coal		Analysis, per cent. (Parr basis)			Davis Plastometer, Temp. at which resistance:				Maximum Resistance, lb.	Damm-Agde Test, Temp. (° C.) of:			Layng-Hathorne Test, Temp. at which resistance:	
No.	Name	Carbon	Hydrogen	Vol. Mat.	Developed	Max.	Min.	Dropped.		Softening	Expansion: Begins	Expansion: Ends	Developed	Dropped
23	Pocahontas	92·4	4·44	15·8	465	468	466	474	3·6	407	462	490	448	547
26	Sewell	90·3	4·94	21·2	414	436	447	468	43·9	378	431	466	415	534
27	Sewell	88·8	5·25	27·4	415	421	431	465	29·7	320	396	430	393	526
4	Davis	88·9	5·48	25·7	411	415	423	427	25·8	355	408	---	412	498
8	Mary Lee	88·2	5·28	30·8	393	393	434	457	49·0	319	380	---	384	461
1	Pittsburgh	86·7	5·75	37·0	387	397	421	443	34·7	291	372	---	395	447
28	Pittsburgh	86·0	5·51	37·4	393	404	421	447	48·5	298	378	---	382	443
22	Pittsburgh	85·2	5·76	40·1	390	400	419	442	34·4	286	376	416	387	439
10	No. 6 Illinois	83·4	5·37	39·4	No resistance					275	414	—	405	540
19	Lower Sunnyside	82·3	5·83	42·9	No resistance					295	394	433	390	443
21	Green River	81·9	5·12	40·1	No resistance					236	398	413	401	450

used. In the Layng-Hathorne (modification of the Foxwell) test the coal mixed with about half its weight of electrode carbon was heated in a vertical tube at a rate of 3·2° C. per min. over the plastic range.

There is obviously a general relationship between the temperature of swelling (Damm-Agde test), the temperature at which the resistance rises in the Layng-Hathorne test, and the temperature, at which resistance develops in the Davis plastometer. Moreover, the temperatures at which resistance began to fall in the Layng-Hathorne and the Davis plastometer tests agree for a number of coals. In all three tests, however, the coal is free to expand and the plasticity ends before it does when swelling is prevented, as it is in coking practice.

K. Gieseler has used a penetrometer and a modified plastometer to measure the plastic range of coal. In his penetrometer (*Glückauf*, 1932, 1102 : 1933, 604), a briquette 7 mm. in diameter was heated in a glass tube. A needle at the end of a rod rested on the briquette and was suitably loaded, the support of the rod passing over a pulley to which a pointer was attached to move over a scale. The temperature of the coal was raised at a rate of 2° C. per minute over the critical range. The "softening-point" was taken to be the temperature at which the needle began to sink into the coal, and the "solidification-temperature" that at which the needle would no longer sink.

In a modification of the Foxwell test, Gieseler passed nitrogen through a vertical column of coal, 10 mm. in diameter and 50 mm. high, resting on a layer of firebrick. In his plastometer (*Glückauf*, 1934, 169), Gieseler used a vertical steel tube, 10 mm. in diameter and 42 mm. long, containing about 2·3 grammes of coal. A stirrer, 1·5 mm. in diameter, had a spiral with only a small clearance with the walls of the tube. The charge of coal was enclosed at the top and bottom by perforated plates and almost filled the container. Gas was discharged through the upper perforated plate to a side exit. The stirrer, mounted on ball bearings, was connected to a weight-pan and the load required to move it was measured. Standardization in absolute units was effected by using liquids of known viscosity. The plastometer was heated at a rate of 3° C. per min. The results of Gieseler's experiments are summarised in Table CXII.

In effect, the "softening-point" as measured by the penetrometer was found to be the same as the temperature at which plasticity was first observed with the plastometer, a surprising result since, as shown by the work of Fieldner and Davis (Table CXI), and by our own work, the softening of coal usually occurs at much lower temperatures than those recorded by Gieseler. The "solidification-temperature" was the same when determined by either the penetrometer or the plastometer method, and was usually near to the temperature of maximum resistance to the passage of gas as recorded

TABLE CXII.—PLASTICITY MEASUREMENTS OF GIESELER.

Coal		Vol. Mat. per cent. on "pure coal" basis	Plasticity begins		Initial Resistance "Foxwell" Test	Solidification-Point		Final low Resistance "Foxwell" Test	Swelling Range		Swelling, per cent.
			Penetrometer	Plastometer		Penetrometer	Plastometer		Begins	Ends	
			° C.	° C.	° C.	° C.	° C.	° C.	° C.	° C.	
Ruhr	A	17·3	463	461	474	515	515	537	468	501	19
	B	18·2	410	413	440	503	503	530	431	485	74
	C	20·7	405	402	405	512	512	527	425	487	97
	D	22·1	380	376	400	502	493	481	391	463	115
	E	23·3	386	380	407	506	500	473	400	471	144
	F	24·3	388	—	400	502	—	470	397	466	137
	G	25·3	390	—	402	510	—	511	405	467	150
	H	25·4	384	—	401	506	—	471	398	466	226
	I	26·0	374	373	385	502	487	472	378	461	260
Lower Silesia	K	24·7	395	390	398	504	503	508	406	444	18
	L	26·9	398	—	405	491	—	494	401	459	15
	M	28·2	389	384	400	505	495	493	398	461	75
	N	34·8	370	367	300	477	475	464	401	425	12
	V	32·6	—	—	—	—	462	—	—	—	—
Upper Silesia	O	29·6	373	370	393	497	490	457	391	461	137
	P	32·5	375	372	400	495	483	453	393	452	76
	R	34·7	382	—	398	472	—	479	400	446	13
	S	35·7	378	378	400	478	468	486	398	450	11
	T	39·2	381	378	400	468	468	467	413	431	20
	U	39·3	388	—	400	456	—	440	404	422	6

by a modification of Foxwell's test. If these results are confirmed by other experimenters, the penetrometer appears to be a useful means of measuring the "solidification-temperature" of coal.

An important feature of Gieseler's results is that he found the same "solidification-temperature" for coal prevented from swelling, in his plastometer, as in his penetrometer, where the coal was free to swell. This apparent confliction with our results is probably due to the fact that, with the small amount of coal and the large stirrer used in Gieseler's plastometer, the formation of coke in an undisturbed state could not occur, the constant stirring allowing the continuous escape of volatile matter, so that the results obtained do not actually represent the behaviour of coal in the plastic state prevented from swelling. It seems to be difficult, if not impossible, to modify the plastometer so as to allow of the undisturbed formation of coke.

Gieseler assigned a maximum plasticity of 200 to coal I of the Ruhr coals, the value falling with decrease in volatile matter and being only 1 for Coal A. For the Upper Silesian coal O a maximum value of 600 was obtained, the other coals of that group having relatively low values. The Lower Silesian coals gave values varying from 14 to 39. A low value for plasticity would make a coal a poor coking coal, but no special advantage appears to attach to a coal of high plasticity. Although Gieseler records temperatures of 460–

500° C. for the "solidification-temperatures" of coals (unless they are of very low volatile matter content) the same order of values as we have suggested for coal prevented from swelling, it must be remembered that Gieseler used a rate of heating of 3° C. per minute as compared with our slow rate of 1° C. per minute. Accepting Gieseler's results as a relative series, it may be noted that solidification tends to end at a lower temperature the higher the volatile matter content (and, in general, the lower the rank) of the coal. Coals of high rank, by remaining plastic to a high temperature, leave less volatile matter to be evolved after plasticity has ceased, and therefore the cokes made from them are less strongly fissured by shrinkage cracks.

H. A. J. Pieters, H. Koopmans and S. W. T. Hovers (*Fuel in Science and Practice*, 1934, **13**, 82) examined the plasticity of coal by measuring the tension on a spring, one end of which was fastened to the spindle of a drum and the other to a needle which could be moved in the coal heated in a cylindrical container below the drum. By moving the drum and allowing the needle to move through the coal when it was plastic, its resistance to movement was measured. In these tests the coal was free to swell. With a rate of heating of 3° C. per minute, these investigators found that, whilst the force required to move the needle through the coal was 1·36 (arbitrary scale) at about 410° C. and fell to under 0·2 about 20° C. higher, the force required increased above about 480° C., until at a temperature between 530 and 540° C. high values were required for movement of the needle. Some results for six vitrains of different rank are recorded in Table CXIII.

TABLE CXIII.—PLASTICITY PHENOMENA OF COAL (PIETERS, KOOPMANS AND HOVERS).

Vitrain No.	Vol. Mat. per cent.	Minimum Resistance *	Temp. Range (° C.) for resistance <25
1	30·6	1·0	97
2	27·5	1·5	114
3	25·8	2·5	117
4	24·3	3·0	107
5	19·3	21·0	25
6	19·9	37·0	0

* Arbitrary scale readings.

The higher the volatile matter the lower the minimum scale reading, or the more plastic the coal. The two vitrains of low volatile matter content did not become as plastic as the others and did not maintain plasticity for long, but the coals of 24·3–27·5 per cent. volatile matter content were markedly plastic over the initial temperature-range.

Despite the inadequacies of many of the methods of measuring plasticity and its duration, it is clear that, to form a coke, coal must become relatively plastic and must maintain this plasticity over a suitable temperature-range. The longer the plastic state is prolonged, the less volatile matter remains to be evolved after solidification. The coals which give the best cokes are not necessarily those which develop the greatest plasticity, although such coals may be the most valuable for blending with others which do not become sufficiently plastic. Coals of low plasticity are either of very low hydrogen or volatile matter content or of low rank. It may be concluded that, when a coal is heated at a rate of 1° C. per minute, its plasticity ends at about 500° C., but that the temperature is higher with more rapid rates of heating. One merit of rapid heating, therefore, is to prolong the plastic period and thus enable as much volatile matter as possible to be evolved before "solidification."

The relationship between the evolution of volatile matter and the persistence of plasticity is important. If a coal is free to swell, it loses volatile matter more readily, and plasticity ends at a lower temperature, than if the coal is confined. If swelling is prevented, as it is in a coke-oven in practice, the volatile matter is almost all retained by the plastic coal until plasticity ceases and is then suddenly released. The continuance of plasticity to a high temperature by preventing a coal from swelling (or by increasing the rate of heating), shows the relationship between plasticity and the primary cause of swelling, namely, gas-pressure. Since the difficulties involved in measuring the plasticity of coal directly under the conditions obtaining in a coke-oven are so great, it is likely that more reliable information can be obtained by a study of swelling phenomena.

The Relationship between the End of Plasticity, the Evolution of Volatile Matter, and the Porosity of Cokes

It has been shown that, when coal is prevented from swelling, there is a sudden evolution of volatile matter at about 500° C. So long as the volatile matter is retained, the pores in the plastic coal must remain closed, but after its evolution the proportion of closed pores is probably small. Some open pores may be re-closed by secondary thermal reactions, such, for example, as involve the deposition of carbon. We have attempted to study the formation of pores in coke made from coal heated at constant volume, in the apparatus illustrated in Fig. 86.

A retort of mild steel of 1-in. internal diameter was fitted with a plunger, having a clearance of $\frac{1}{64}$-in., one end of which was fixed in a socketed flange. One end of the retort was closed by a hexagonal nut, the original intention of which was to provide for the ready removal of the coked charge. Actually, the nut was

found to be essential to allow of the release of some of the gas-pressure during the carbonisation.*

Cokes were prepared at constant volume by heating charges of coal to a series of temperatures, at a rate of rise of temperature of 1° C. per minute, and cooling the retort rapidly in a stream of water as soon as the desired temperature had been attained. The percentages of open and closed pores in the cokes were determined by the methods described on p. 94. The results obtained are recorded in Table CXIV.

TABLE CXIV.—PERCENTAGES OF OPEN AND CLOSED PORES IN COKES MADE AT CONSTANT VOLUME.

Temp. ° C.	Vol. Matter in Product, per cent.	Apparent Sp. G.	Real Sp. G.	Total Pores, per cent.	Open Pores, per cent.	Closed Pores, per cent.
350	34·1	*	1·31	*	*	*
400	30·5	1·29	1·33	3	1	2
450	29·8	0·89	1·34	33	17	16
500	19·0	0·75	1·40	46	42	4
550	18·8	0·70	1·40	51	46	5

* Not determinable since a homogeneous coke is not obtained.

These results show that, with coal prevented from swelling, there is but a small evolution of volatile matter up to 450° C., with a marked increase in the amount evolved between 450° and 500° C. The records of apparent specific gravity are of no significance until the coal particles had agglomerated, at a temperature exceeding 400° C. It will be noted that the percentage of closed pores decreased from 16 at 450° C. to 4 at 500° C., a result in agreement with the presumed rupturing of closed pores at the temperature (490° C.) of sudden evolution of volatile matter.

Attempts were now made to determine more closely the temperature at which the marked change in the proportion of closed pores occurred, and two further tests were made at 450° and 460° C., with the following result:

	450° C.	460° C.
Apparent Sp. Gr. . .	0·85	0·81
Total pores, per cent. .	38	42
Open pores, per cent. .	24	39
Closed pores, per cent. .	14	3

* If release of gas were allowed only by the clearance around the plunger the plunger rod was buckled and the retort itself distorted by the pressure developed. Although gas-tight at low pressures, the nut allowed release at high pressures.

This result was rather surprising, for we did not expect there to be a marked reduction in the proportion of closed pores at a temperature much below that at which there was a sudden evolution of volatile matter. An explanation is afforded by the following observations. The retort containing a coke prepared at 470° C. was cooled in air (in a bell-jar) instead of in a stream of water. After cooling had been in progress four minutes, vapour was observed to issue in a rapid stream at the nut closing the top of the retort and globules of tar condensed on the bell-jar. In further tests, this phenomenon was only observed when the cokes had been prepared between 450° and about 500° C. Cooling was then accompanied by an evolution of tar-vapour, and the higher the temperature (up to 500° C.) the shorter was the interval before the evolution occurred. When the cokes were prepared at temperatures exceeding 500° C., a sudden evolution of gas and vapour occurred during the heating.

It would seem, therefore, that if the temperature to which the retort was heated exceeded 500° C., the coal passed through the plastic stage and solidified, whereas if the temperature was less than about 500° C. the coal remained plastic until it began to cool. On cooling to a sufficient degree, the coke solidified and there was then a sudden evolution of volatile matter which disrupted most of the closed pores. The proportion of closed pores existing in cokes prepared at a temperature between 450° and 500° C., and maintained at that temperature, is therefore not the same as when the coke is cooled.

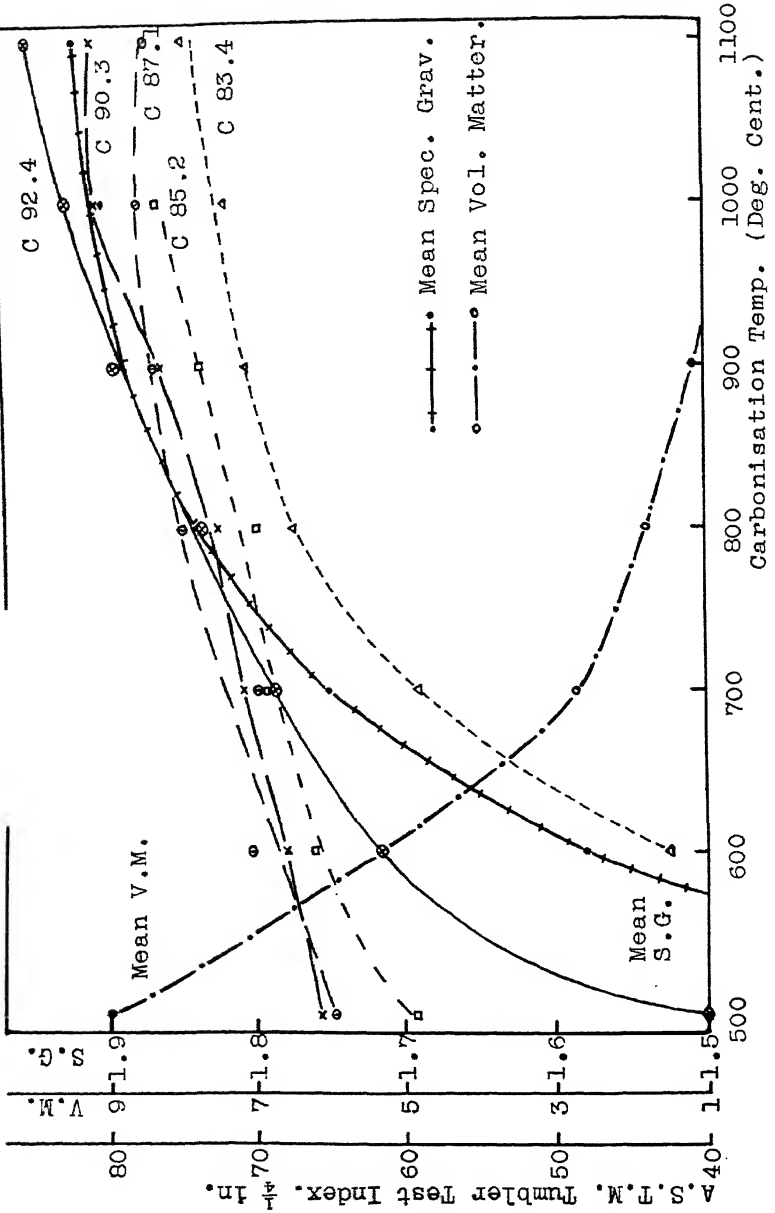
CHAPTER XVII

THE FORMATION OF COKE FROM SEMI-COKE

When the plasticity of coal has ended at a temperature of, say, 500° C., the subsequent changes are concerned only with (1) the hardening of the cell walls and (2) the formation of fractures. The changes which take place may be illustrated by values obtained by the U.S. Bureau of Mines in the "B.M.–A.G.A." test, in which charges of about 100-lb. with a bulk density of 50 lb. per cu. ft. were placed in a steel container and carbonised at various temperatures between 500° and 1100° C. The rate of heating through the plastic stage was about 7° C. per minute for tests at 900° C., this fast rate of heating giving results for poor coking coals which could not be obtained in large-scale practice. Making allowance for this, and for the fact that the volatile matter recorded as remaining in the charge at the end of the heating was rather low because the charges were not quenched but allowed to cool, some of the changes which take place may be illustrated in Fig. 106. This shows the ¼-in. "tumbler-test" index (A.S.T.M. apparatus using a 20-lb. sample) compared with the mean real specific gravity and the mean volatile matter content of seven cokes made from coals covering a wide range of rank. The analyses of the coals are recorded in Table CXV.

Table CXV.—Analyses of some Coals Examined in the B.M.–A.G.A. Test.

	Coal	Air-dry Basis		Parr Unit Coal Basis:			
		Moist.	Ash	C.	H.	Volatile Matter	Cal. Val., B.Th.U. per lb.
23	Pocahontas (W.Va.)	0·8	4·8	92·4	4·44	15·8	15,840
26	Sewell, W.Va. . .	1·3	2·1	90·3	4·94	21·8	15,790
27	Sewell, W.Va. . .	1·9	2·4	88·8	5·25	27·4	15,700
18	Pratt, Ala. . .	3·0	3·1	87·1	5·52	33·2	15,520
22	Pittsburgh, W.Va. .	1·9	6·0	85·2	5·76	40·1	15,300
10	No. 6, Illinois . .	7·9	12·3	83·4	5·37	39·3	14,660
21	Green River, Ky. .	10·1	6·7	81·9	5·72	42·6	14,560



106.—Increase in Surface Hardness of Cokes from five Coals of Different Carbon-Content (C%) on Carbonising at Temperatures of 500 to 1100° C. Mean Specific Gravities and Mean Volatile Contents of the Cokes.

The Loss of Volatile Matter

Mean values are used in Fig. 106 for volatile matter and real specific gravity since the individual values differ but little for the cokes from the different coals. The volatile matter drops rapidly between 500° and 700° C. and then more slowly to 1100° C. The real specific gravity rises rapidly between 500° and 800° C. and then rises more slowly. The ¼-in. tumbler index, which is a measure of the abradability (surface hardness) of the coke, rises with increase of temperature of carbonisation. For the cokes from the three coals of 90·3 to 85·2 per cent. carbon-content it rises gradually over the whole range of temperatures used, but with the cokes from the high-rank Pocahontas and the low-rank Green River coals, which do not exhibit much plasticity in other tests, the rise is steep over the lower part of the temperature-range and is then more gradual. For these five coals the hardness of the cell-walls at 900° C. and higher varies in the same sense as the rank of the coals.

The results in Fig. 106 illustrate the well-known "hardening" process which takes place when semi-coke is heated to a high temperature before it is discharged. The extent of the hardening process is not proportional to the loss of volatile matter, or to the increase in real specific gravity, but varies with the rank of the coal used.

Table CXVI.—Ultimate Analysis (dry, ash-free) of Cokes made at Different Temperatures.

Coal No.	Coals			500°			600°			700°		
	C.	H.	O.	C.	H.	O.	C.	H.	O.	C.	H.	O.
23	91·8	4·46	2·0	92·1	3·5	2·3	93·7	2·7	1·6	95·5	1·8	1·0
26	89·7	4·92	2·8	90·9	3·6	2·7	92·8	2·5	1·9	94·8	1·5	1·2
27	88·4	5·22	4·2	91·4	3·2	2·8	93·3	2·3	2·1	95·0	1·6	1·3
18	86·8	5·50	5·3	90·0	3·3	4·2	92·9	2·4	2·4	94·6	1·7	1·5
22	84·5	5·74	7·3	89·5	3·4	4·4	92·2	2·6	2·5	94·2	1·5	1·4
10	82·1	5·52	9·6	88·5	3·5	5·4	91·2	2·5	3·4	93·5	1·7	2·2
21	80·3	5·62	9·3	88·2	3·4	4·2	91·1	2·5	2·0	93·2	1·5	1·1
Mean				90·1	3·4	3·7	92·4	2·5	2·3	94·4	1·6	1·4
		800°			900°			1 000°			1 100°	
23	96·9	1·2	0·2	97·5	0·7	0·1	97·9	0·5	0·1	98·2	0·3	0·1
26	95·0	1·2	1·1	96·1	0·9	0·3	96·8	0·5	0·3	97·2	0·4	0·3
27	95·8	1·3	0·7	96·9	0·7	0·2	97·4	0·5	0·3	97·6	0·3	0·8
18	95·7	1·1	0·9	96·8	0·7	0·1	97·0	0·5	0·5	97·4	0·3	0·8
22	94·6	1·4	1·6	96·2	0·9	0·5	97·0	0·6	0·3	97·6	0·3	0·3
10	94·8	1·1	1·5	96·2	0·6	0·9	96·9	0·6	0·4	97·6	0·4	0·3
21	94·2	2·1	0·5	94·8	0·8	0·1	95·8	0·5	0·1	96·8	0·3	0·1
Mean	95·3	1·2	0·9	96·4	0·8	0·3	97·0	0·5	0·3	97·5	0·3	0·4

Note.—The sulphur-contents of the coals were only 0·5–0·8 per cent., with the exception of No. 21 which contained 3·0 per cent. (dry, ash-free basis).

It is noteworthy (see Table CXVI) that, despite the differences in hydrogen-contents of the original coals, at 500° C. all the cokes are of approximately the same hydrogen-content, whilst at higher temperatures the cokes made from the different coals differ but little in the amount of hydrogen they contain. The mean values for hydrogen for the seven series of cokes made at different temperatures are graphed in Fig. 107, which shows that at 700° C. there is a critical stage, the elimination of hydrogen at higher temperatures being much slower.

There are, however, differences in the carbon-contents of the cokes made at 500° and at higher temperatures. This is indicated in Fig. 108, which suggests that original differences in the rank of the coals are maintained in the cokes up to a carbonisation-temperature of 700° C., above which they become less obvious, except for the coals of highest and lowest rank. The mean value of the carbon-content in Fig. 107 shows a break at 700° C. as does that of the hydrogen-content.

The changes in oxygen-content are illustrated in Fig. 109, showing that generally the coals of higher oxygen-content retain more oxygen in their cokes. The mean changes in nitrogen-content are shown in Fig. 107, the behaviour being similar for all the coals. There is a slow reduction in nitrogen up to 900° C., and subsequently a more rapid drop. The sulphur remains fairly constant from 500° to 1100° C.

The chief chemical change which occurs, therefore, is the elimination of oxygen and hydrogen. By calculating the mean yields of liquor per ton of dry, ash-free coal for the series, an increase of 1·0 and 0·1 per cent. is found between 500°–600° C. and 600°–700° C., but there is a reduction in the yield of liquor at higher temperatures owing to its decomposition. By plotting the percentage yield by weight of carbon monoxide at different temperatures a regular increase over the whole range of temperatures is found, equivalent to an increase of 0·7 per cent. carbon monoxide (or 0·4 per cent. oxygen) per 100° C. rise in temperature. Loss of oxygen as carbon monoxide may, therefore, be assumed over the whole range of temperatures, with a loss of oxygen as water over the lower range of temperatures.

To allow for loss of hydrogen it is not possible to assume that much is evolved as methane, which has the smallest carbon to hydrogen ratio of the hydrocarbons, for the calculated increase in carbon-content is too low. It is necessary to assume that there is a loss of free hydrogen with some methane, the proportion of methane falling up to 700°, above which temperature no evolution of methane can be assumed.

An example will make this clear.

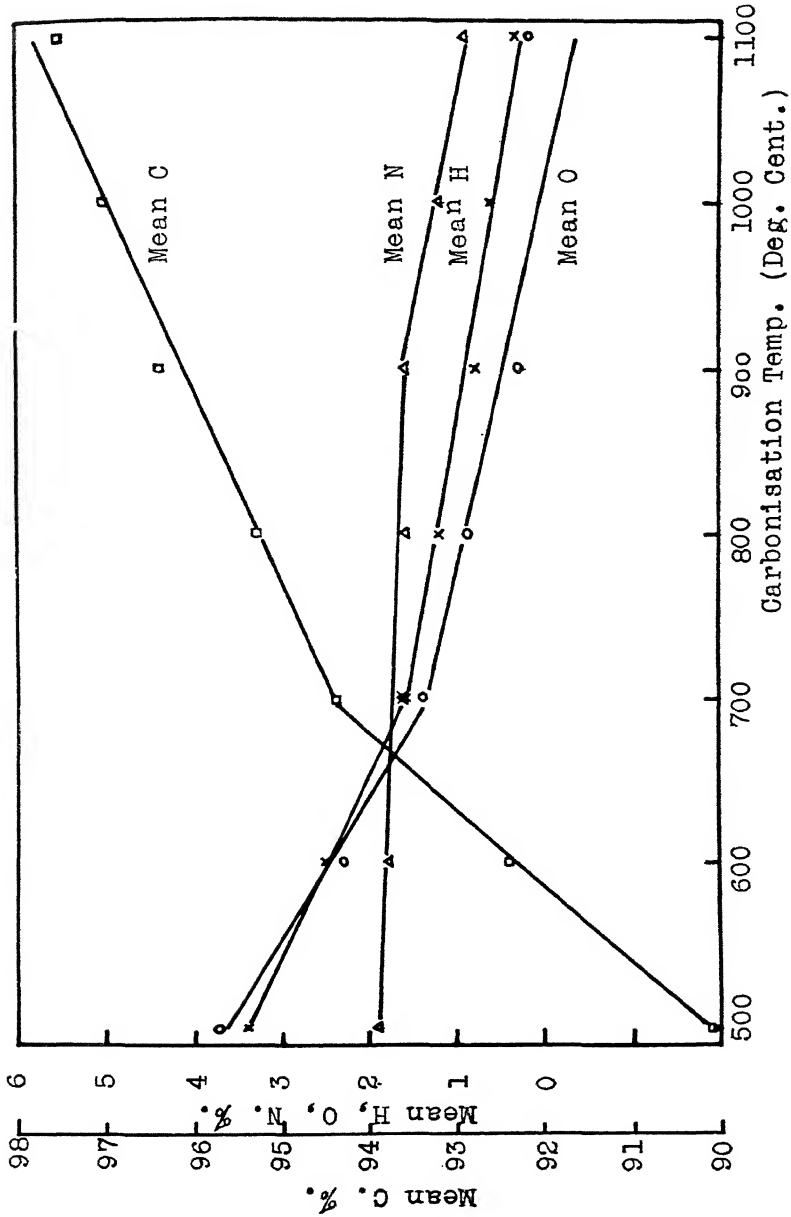


FIG. 107.—The Mean Changes in Composition of Cokes between 500 and 1100° C. made from Coals of Table CXVI.

	Carbon	Hydrogen	Oxygen
Mean composition of coke at 500° C.	90·4	3·4	3·7
Assume loss of 0·4 O_2 as CO, then loss of C and O_2 is: ..	0·3	—	0·4
Assume loss of 0·9 O_2 as H_2O, then loss of H_2 and O_2 is: ..	—	0·1	0·9
Assume loss of 0·6 H_2 as H_2, then loss of H_2 is:	—	0·6	—
Assume loss of 0·3 H_2 as CH_4, then loss of C and H_2 is: ..	1·1	0·3	—
Residual composition with loss of 3·7 per cent.	89·0	2·4	2·4
Calculated percentage composition of residue (at 600°) ..	92·4	2·5	2·5
Actual percentage composition of residue (at 600°)	92·4	2·5	2·3

The changes in composition of the residues are accounted for approximately on the following assumptions:

500–600° C.	Loss of 0·9 O_2 as H_2O, 0·4 O_2 as CO, 0·6 H_2 as H_2, 0·3 H_2 as CH_4.
600–700° C.	Loss of 0·9 O_2 as H_2O, 0·4 O_2 as CO, 0·6 H_2 as H_2, 0·3 H_2 as CH_4.
700–800° C.	Loss of 0·4 O_2 as CO, 0·4 H_2 as H_2.
800–900° C.	Loss of 0·4 O_2 as CO, 0·4 H_2 as H_2.
900–1000° C.	Loss of 0·3 O_2 as CO, 0·3 H_2 as H_2.
1000–1100° C.	Loss of 0·3 H_2 as H_2.

With these assumptions the calculated and the actual compositions of the coked residues are compared in Table CXVII.

TABLE CXVII.—COMPARISON OF CALCULATED AND ACTUAL COMPOSITIONS OF COKED RESIDUES.

	Calculated			Actual		
	Carbon	Hydrogen	Oxygen	Carbon	Hydrogen	Oxygen
500°	—	—	—	90·1	3·4	3·7
600	92·2	2·5	2·5	92·4	2·5	2·3
700	94·4	1·56	1·25	94·4	1·6	1·4
800	95·4	1·17	0·86	95·5	1·2	0·9
900	96·2	0·78	0·47	96·4	0·8	0·3
1000	97·0	0·49	0·18	97·0	0·5	0·3
1100	97·3	0·20	0·19	97·5	0·3	0·2

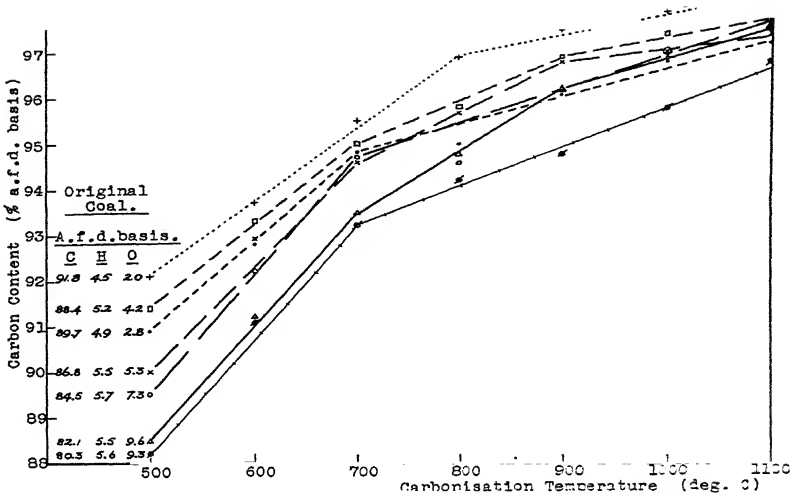


FIG. 108.—Individual Changes in Carbon Contents of Cokes between 500 and 1100° C. made from Coals of Table CXVI.

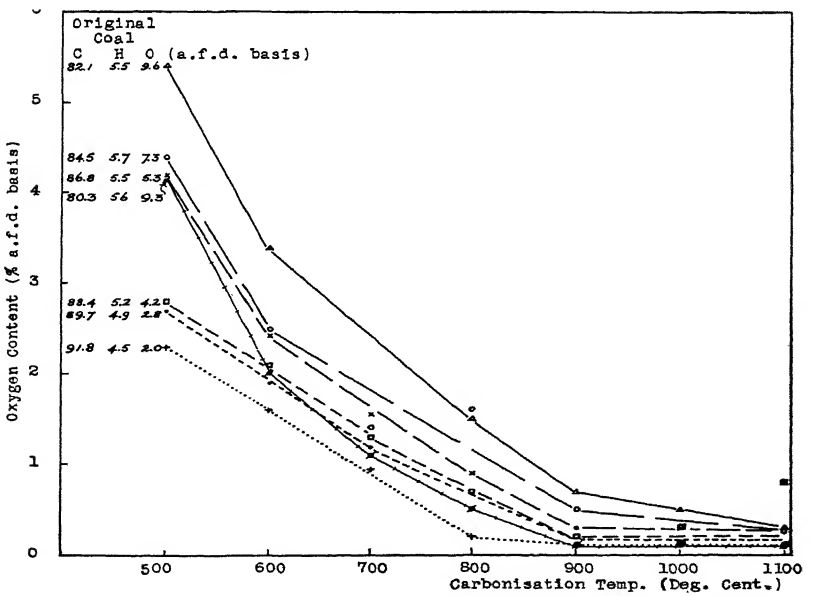


FIG. 109.—Individual Changes in Oxygen Contents of Cokes between 500 and 1100° C. made from Coals of Table CXVI.

These changes may be summarised as showing between 500° and 700° C. a rapid fall in the oxygen and hydrogen content due to the loss of water and of hydrogen associated with carbon in the atomic ratio C : 10 H ; and at 700° C. and upwards a gradual loss of oxygen and hydrogen as carbon monoxide and free hydrogen, the amount of carbon monoxide evolved falling when the oxygen remaining is under 0·5 per cent. The amounts of paraffin hydrocarbons evolved from the coal may, however, appear to increase at temperatures above 700° owing to cracking of tar previously formed.

TABLE CXVIII.—WEIGHT PER CENT. OF DIFFERENT GASES ON HEATING COALS TO DIFFERENT TEMPERATURES (° C.).

Coal	C.	H.	Vol. Mat.*	Gas	500	600	700	800	900	1000	1100
Anthracite	93·5	3·17	12·5	H_2		0·2	0·4	0·9	1·2	1·5	1·8
				CH_4, C_2H_6		0·7	1·2	2·1	2·4	2·2	2·2
				CO		0·1	0·3	1·1	2·2	2·8	5·0
D.	91·4	4·27	19·0	H_2	0·0	0·2	0·4	1·0	1·4	1·8	2·2
				CH_4, C_2H_6	0·9	2·2	3·4	4·5	4·2	4·0	3·8
				CO	0·1	0·3	0·4	1·5	2·9	3·7	4·6
C.	86·4	4·08	31·5	H_2	0·0	0·2	0·4	0·9	1·3	1·6	1·7
				CH_4, $_2H_6$	2·6	5·2	5·3	5·6	5·8	5·3	4·9
				CO	0·2	0·7		2·5	3·6	5·0	5·1
A.	83·0	5·61	38·8	H_2	0·0	0·2	0·4	0·9	1·3	1·5	1·7
				CH_4, $_2H_6$	2·7			5·9	5·4	5·2	5·6
				CO	0·2	0·9	1·2	3·2	4·6	5·7	6·5

* At 1100° C., per cent. on dry, ash-free coal.

Although the mean value for the carbon-content of all the cokes produced at different temperatures shows a break at 700° C., the curves for the cokes obtained from individual coals show breaks between 700 and 800° C. Such a change in the composition of the products of distillation was first pointed out by Burgess and Wheeler (*J. Chem. Soc.*, 1910, **97**, 1917; *Fuel in Science and Practice*, 1925, **4**, 208), who heated coals in a small platinum retort (2 grammes of coal being mixed with 3 grammes of sand) at temperatures between 500 and 1100° C. and analysed the gaseous products. Some secondary decomposition of tar probably occurred owing to the position of a tar scrubber within the retort. It was observed that the total amount of methane obtained did not increase above 750° C.,